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ULTRAFILTRATIVE DEWATERING OF SPENT POWDERED CARBON

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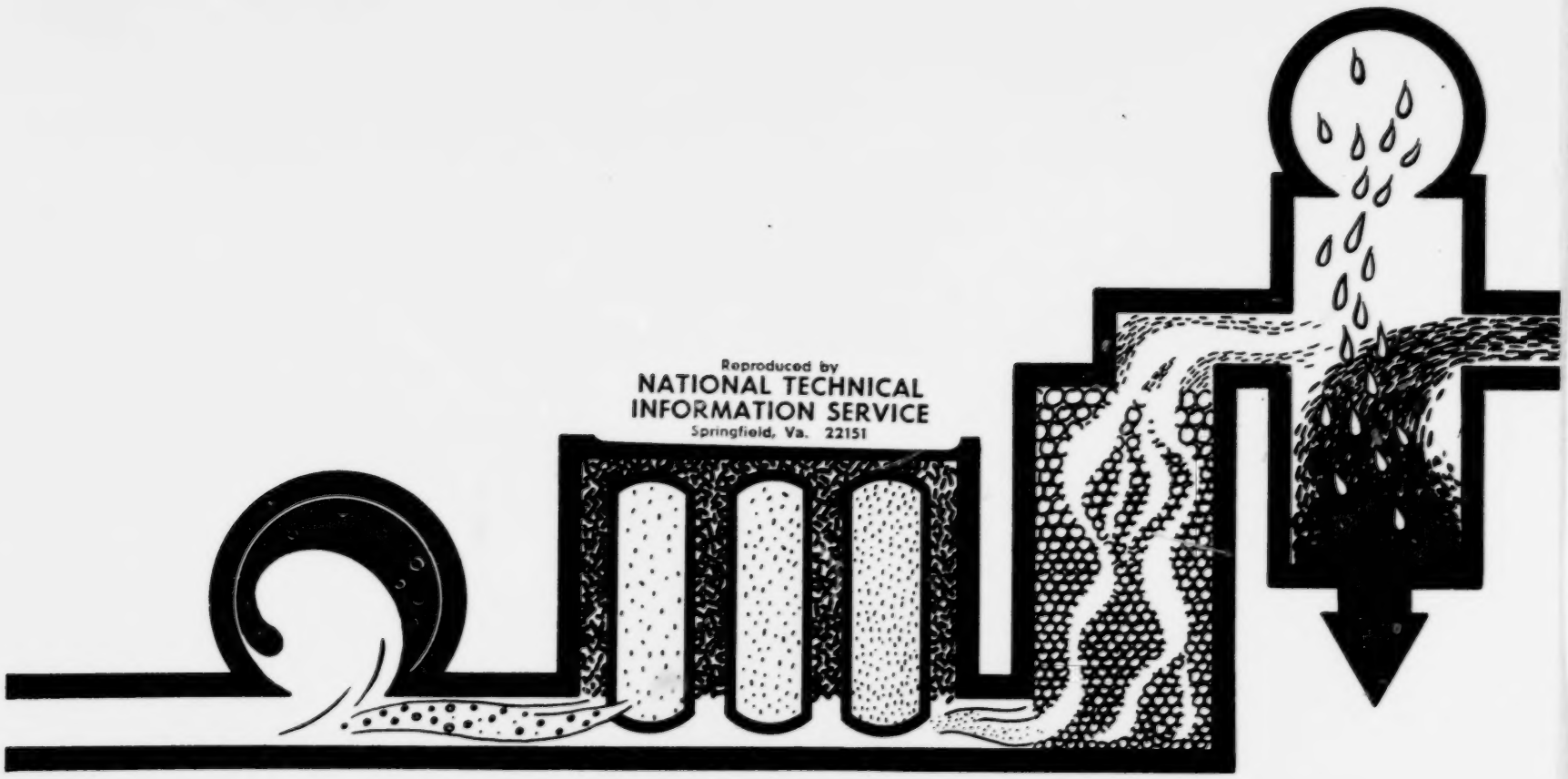

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27 Abstract Spent powdered activated carbon from secondary effluent treatment has been successfully dewatered by continuous membrane ultrafiltration. 5 and 10% carbon solids concentration slurries, using a laboratory size thin channel(0.1 ft^2 membrane area), have been dewatered by continuous ultrafiltration to solids levels of 25 to 30%. Dewatering rates ranged between 50 and 100 gal/ft^2 day at transmembrane pressures of from 10 to 50 psi during runs as long as 9 days, and product water was invariably free of any suspended carbon solids. The cost of membrane ultrafiltration depends upon the membrane life, and at membrane flux rates of 50 gal/ft^2 day the cost of dewatering carbon from 10 to 20% solids is $0.0015 per pound of carbon with a membrane life of six months, and $0.0035 per pound with a membrane life of only one month. At the present no conclusive data on membrane life is available, but estimates of between 1 and 6 months are quite common and seen to be reasonable. (Lowry-Texas)

(WRSIC abstract)

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ULTRAFILTRATIVE DEWATERING OF SPENT POWDERED CARBON

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TABLE OF CONTENTS

	page
SUMMARY	
INTRODUCTION AND BACKGROUND	1
MATERIALS AND EQUIPMENT	3
Spent Carbon Concentrates	3
Equipment	7
Membranes	16
DEWATERING RUNS	21
Virgin Carbon	21
Comparison of Dewatering Cells Using Lebanon Samples	23
Thin Channel Runs Using Brockton Samples With the Gear Pump	28
Thin Channel Runs With the Moyno Pump	32
Long Term Dewatering Runs	34
Effect of Operating Conditions on Flux Rates	40
RHEOLOGICAL STUDIES	46
Low Shear Rate Experiments	46
High Shear Rate Experiments	48
Effect of Dispersants	50
PROPOSED PLANT SCALE PROCESS	55
General Description	55
Economics	56
RECOMMENDATIONS	66

SUMMARY

The feasibility of dewatering spent powdered carbon from secondary effluent treatment by membrane ultrafiltration has been demonstrated. Spent carbon slurries containing between 5 and 10% carbon solids have been dewatered by cortinuous membrane ultrafiltration in a laboratory size thin channel cell (0.1 ft^2 membrane area) to solids levels of 25 to 30%. Typical dewatering rates range between 50 and 100 gfd at transmembrane pressures of from 10 to 50 psi in runs of up to 9 days duration. The product water from all runs was invariably free of any suspended carbon solids.

The economics of dewatering carbon via ultrafiltration are attractive if membrane replacement is infrequent. Using estimation procedures recommended by the Office of Saline Water, the cost of dewatering carbon from 10 to 20% solids assuming membrane flux rates of only 50 gfd, ranges from $0.0015 per pound of carbon for a membrane lifetime of six months to $0.0035 per pound of carbon for a membrane lifetime of 1 month.

INTRODUCTION AND BACKGROUND

The efficacy of activated carbon as an adsorbent in waste-water renovation is known to be enhanced if the carbon is introduced into the water in finely divided particulate form. A major limitation to the use of fine-particulate carbon in this application is the difficulty of recovering the solid from the liquid in sufficiently high concentration to render its final dehydration and thermal regeneration economic.

Efforts to develop means of recovering the carbon have been directed toward addition of polymeric flocculants to promote sedimentation. Such addition typically results in a 5-10% carbon solids slurry; this slurry may then be centrifuged to increase its solids content to between 20 and 25%. The concentrate is then thermally regenerated. A less costly and more efficient method of dewatering the slurry is highly desirable.

The recent development of high-flux, low-pressure ultrafiltration membranes and devices which are quantitatively retentive for colloidal materials and which are not plugged or fouled by the solids they retain, provides a very promising new approach to carbon-slurry concentration. These systems have been successfully utilized, for example,

(1) To concentrate polymer latices to ca. 60% polymer solids without significant sacrifice in ultrafiltration rate, yielding concentrates which are stable and of relatively low viscosity.

(2) To concentrate thixotropic, small particle size clay suspensions at 6% solids to 12-18% solids also without significant sacrifice in ultrafiltration rate.

Such slurries, which become pseudoplastic and thixotropic at high solids concentrations, can be effectively dewatered by ultrafiltration because their weak network structure is destroyed in the shear field transversing the surface of the ultrafiltration membrane. While the particle-particle contacts

are broken, dewatering takes place driven by the hydraulic pressure drop across the membrane. In addition, the membrane surface is also kept clear by the high fluid velocities.

By analogy to latex and clay dewatering, the utility of ultrafiltration for concentrating fine-particle carbon slurries can be determined by subjecting representative carbon slurries to membrane-ultrafiltration at pressures between 10 and 50 psi, and determining the fractional water removal possible without producing unmanageably high consistency or thixotropy in the concentrate.

Although the addition of polymeric flocculants is necessary for concentrating the carbon slurries by coagulation-flocculation, it is possible that these flocculants will impede the ultrafiltration process by reducing the effectiveness of the shear field in separating the individual carbon particles. Assuming such to be the case, the effect of various chemical deflocculants added prior to ultrafiltration should be evaluated. These deflocculants perform their function by preferentially adsorbing on the surface of the carbon particles, thus detaching the adsorbed moieties of the polymeric flocculant. Typical deflocculants include the polyphosphates, silicates, polycarboxylates, and lignosulfonates.

MATERIALS AND EQUIPMENT

Spent Carbon Concentrates

Sources

During the course of the program several samples of spent carbon concentrate were obtained from the FWPCA's Advanced Waste Treatment Pilot Plant at Lebanon, Ohio. These concentrates were prepared from secondary effluent treated with 200 mg/l Aqua Nuchar A* activated carbon powder and 2 mg/l Primafloc C-7** polymeric flocculant. The time span between the preparation of these concentrates at Lebanon and their processing in the dewatering apparatus in Lexington was typically between 1 and 2 weeks. Although the solids content of the Lebanon slurries varied little from 10%, their fluidity and ease of dewatering varied considerably, as noted in the description of each run.

Most of the spent carbon used in this program was prepared from secondary effluent at the treatment plant in Brockton, Massachusetts. The carbon concentrates from Brockton were usually dewatered within 1 to 3 days of preparation and tended to be more uniform in their dewatering properties.

The Brockton Water Pollution Control Facility, is one of the few relatively modern municipal treatment plants in the Boston area with both primary and secondary treatment. The plant was completed in 1964 and is currently operating at 9.5 mgd out of a design capacity of 12 mgd. Although the waste is largely domestic in origin, a considerable proportion of industrial waste (particularly from tanneries) is in the system from time to time. Sampling during these periods was avoided.

*West Virginia Pulp & Paper Company

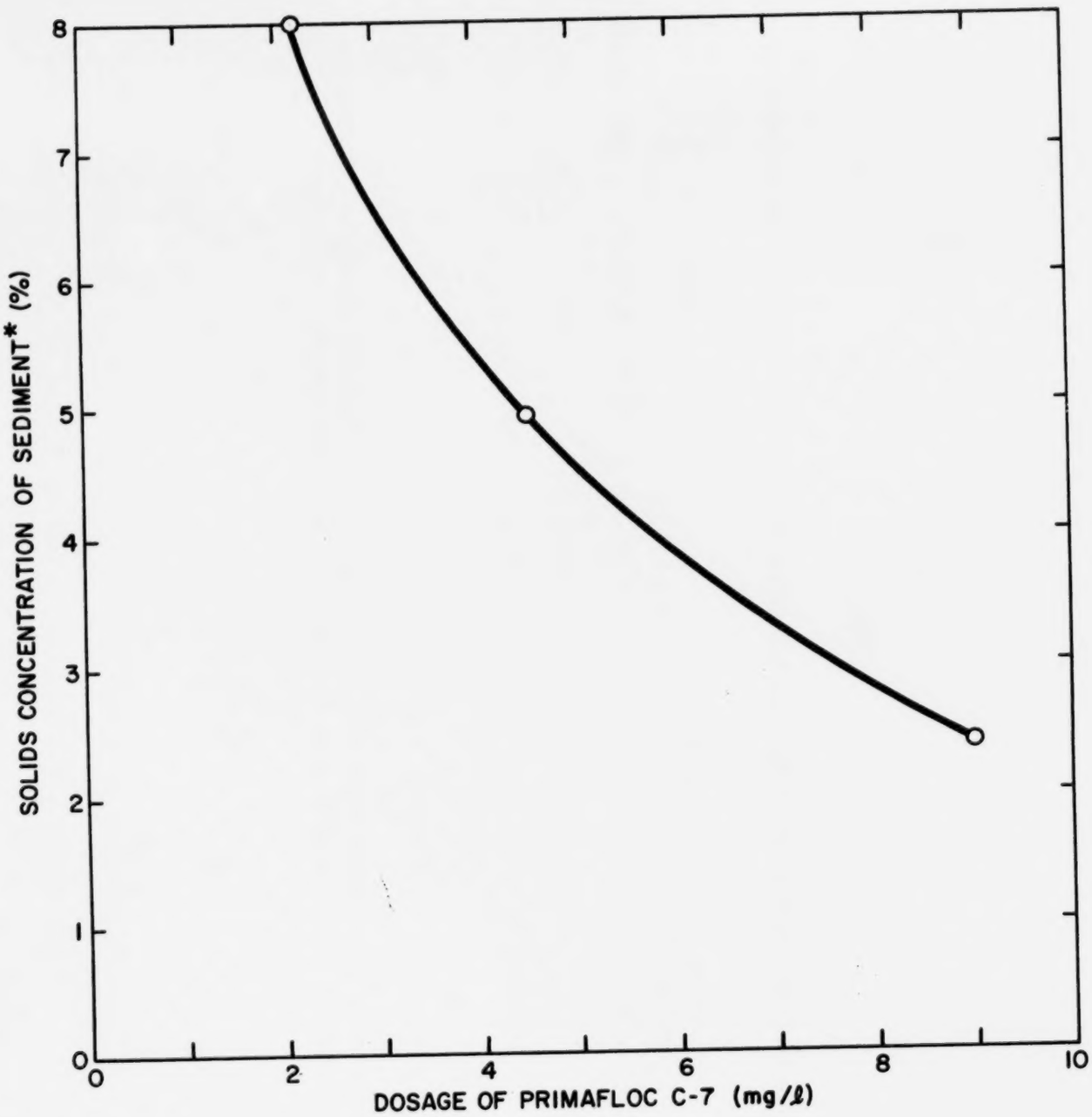
**Rohm & Haas Company

To prepare concentrates of spent carbon from fresh secondary effluent, a 500 gal steel tank was installed next to the Final Settling Tank. The secondary effluent was pumped directly to the tank from the overflow of the secondary settling tank. Aqua Nuchar A previously prepared as a 10% concentrate in water was added with stirring. The carbon was always added at the 200 mg/l dosage level. A 2% concentrate of Primafloc C-7 at 3-5 mg/l dosage was added over a period of 30 minutes to settle the carbon. After 2 hours, most of the carbon floc had settled. The clear supernatant was decanted by siphoning. The spent carbon concentrate plus some unavoidable supernatant, was then drained from the bottom of the tank. After overnight storage in the laboratory, the carbon usually occupied about 2 gallons, corresponding to a carbon solids content of about 5%, which was used for the dewatering experiments.

Effects of Flocculant Dosage

A Primafloc C-7 concentration of 2 mg/l is employed at Lebanon to obtain a spent carbon concentrate of approximately 10% solids. However, preliminary laboratory flocculation experiments at Amicon indicated that a 2 mg/l flocculant dosage would require an impractically long settling time in the 500 gallon "batch" tank in Brockton.

In a typical experiment, a 600 ml sample of Brockton secondary effluent (100 mg/l COD) was mixed with 200 mg/l carbon and after preliminary settling the carbon was poured into a 250 ml graduated cylinder. At 2.2 mg/l of C-7, greater than 4 hours were required to obtain a well-defined sediment. After settling overnight, the sediment was determined to contain 8.2% solids. As shown in Table I and Figure 1, further increasing the flocculant dosage reduced the settling time and the solids concentration. A nominal 4 mg/l C-7 dosage was therefore selected for use at Brockton. This 4 mg/l dosage typically resulted in 5% solids concentrates after 2 hours settling.



*SEDIMENT OBTAINED FROM SECONDARY EFFLUENT (100 mg/ℓ COD) CONTAINING 200 mg/ℓ AQUA NUCHAR A

FIGURE 1. SOLIDS CONCENTRATION OF SEDIMENT VS. PRIMAFLOC C-7 DOSAGE

Table I

Effect of Flocculant Dosage on Settling Time and Sediment Concentration

Secondary Effluent COD, mg/l	Aqua Nuchar A mg/l	Primafloc C-7 mg/l	Settling Time	Appearance of Supernatant	% Solids in Sediment
100	200	9	20 min	clear	2.5%
100	200	4.5	2-3 hrs	clear	5.0%
100	200	2.2	overnight	black*	8.2%

*Supernatant contained sufficient carbon to be black, opaque. Demarcation between supernatant and sediment was consequently diffuse.

Additional information on the effect of the flocculant and carbon concentrations on the solids content of the sediment was obtained later in the program on a poor quality secondary effluent with an unusually high (410 mg/l) COD. This high COD content was due to a foaming problem in the Brockton plant. The COD could be reduced to 136 mg/l after filtration through a 6 inch sand column. A combination of 4 mg/l C-7 and 200 mg/l Aqua Nuchar were insufficient to give the desired rate of settling and a clear supernatant for this effluent. Flocculation tests showed that this material needed higher carbon and flocculant dosage levels. The results are shown in Table II and Figures 2 and 3.

Equipment

Three types of cells were used to dewater carbon concentrates in this program. These were a stirred batch cell, a flow-through, short path, medium channel cell and a flow-through, long path, thin channel cell.

The Batch Cell

An Amicon Model 400 batch cell was used to determine the ultimate level of dewatering possible with a virgin carbon slurry and in some of the preliminary runs on the Lebanon concentrate. This cell, shown in Figure 4, has a capacity of about 400 ml. The feed may be pressurized at up to 50 psi. This cell is equipped with a magnetic stirring bar located near the surface of the membrane in order to minimize the local accumulation of retained solids. Liquid that passes through the membrane is collected in channels beneath the porous support and exits through the ultrafiltrate port shown in the figure.

The Short Channel Cell

A Dorr-Oliver short channel cell was used in some of the preliminary runs with the Lebanon concentrate. In this cell (See Figure 5) the feed stream is passed along a channel about 4 in. long by 1 1/2 in. wide and 1/8 in. deep. The channel is bounded on one side by a membrane, beneath which is located the porous membrane

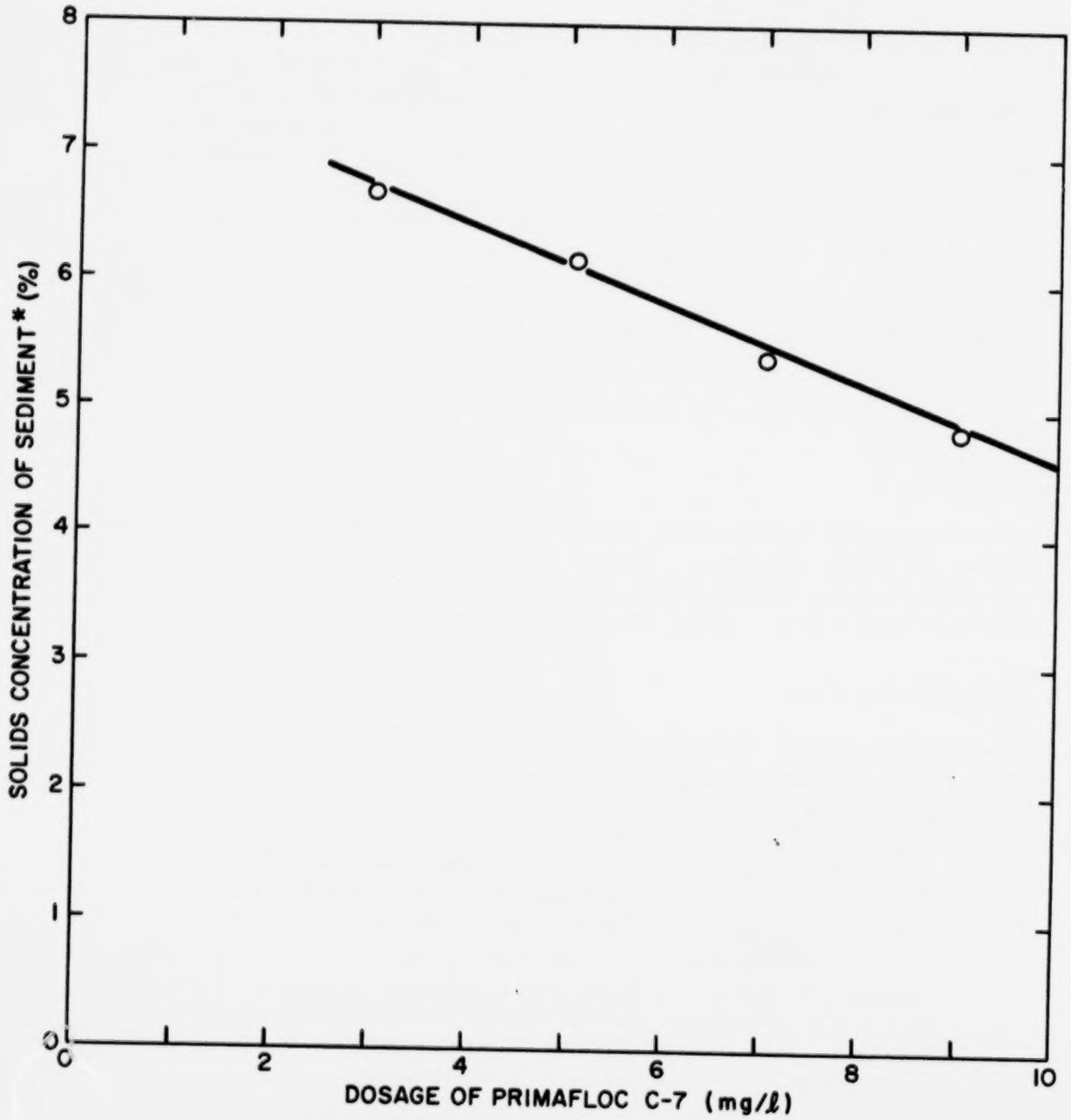


FIGURE 2. SOLIDS CONCENTRATION OF SEDIMENT VS. PRIMAFLOC C-7 DOSAGE

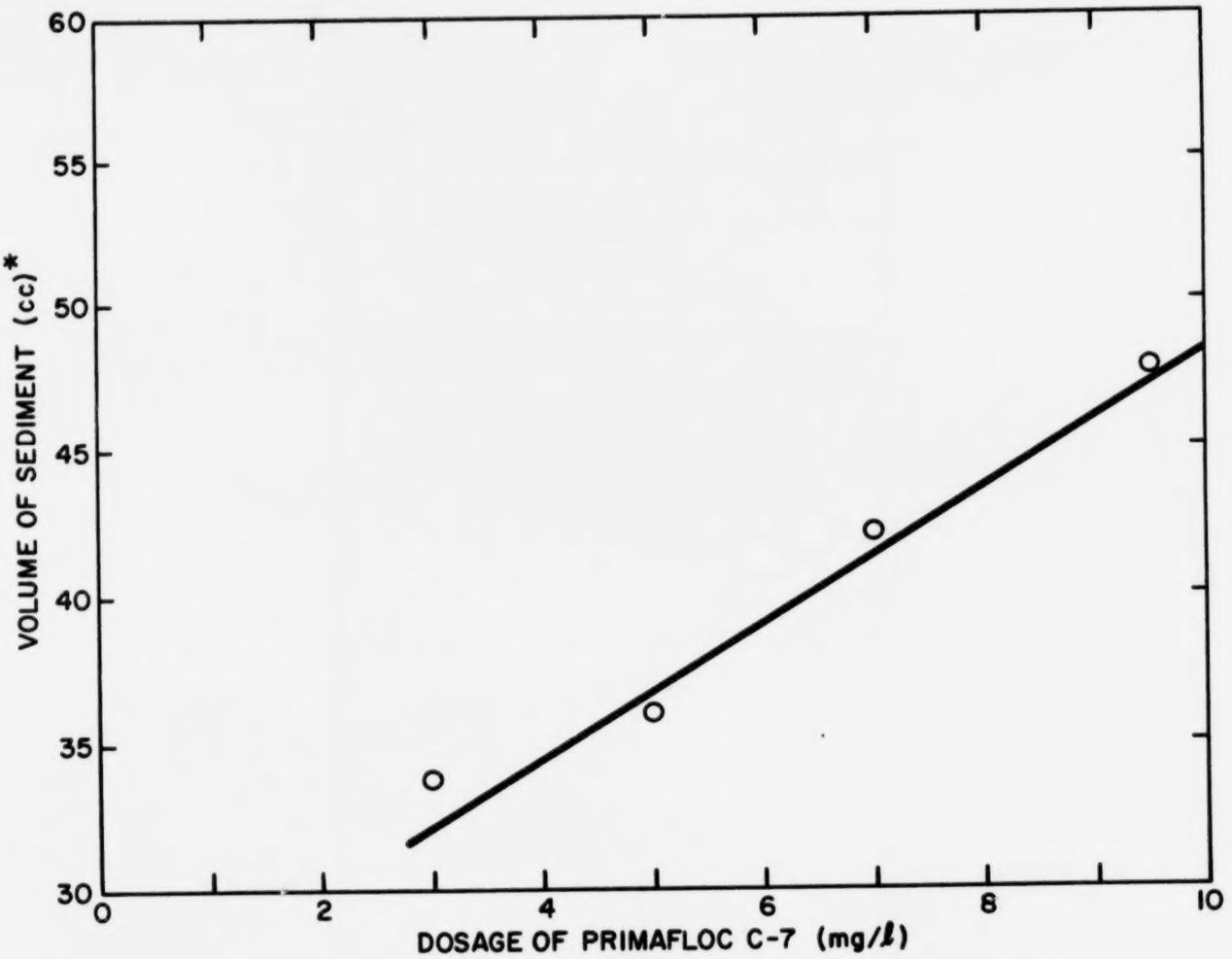


FIGURE 3. SEDIMENT VOLUME VS. PRIMAFLOC C-7 DOSAGE

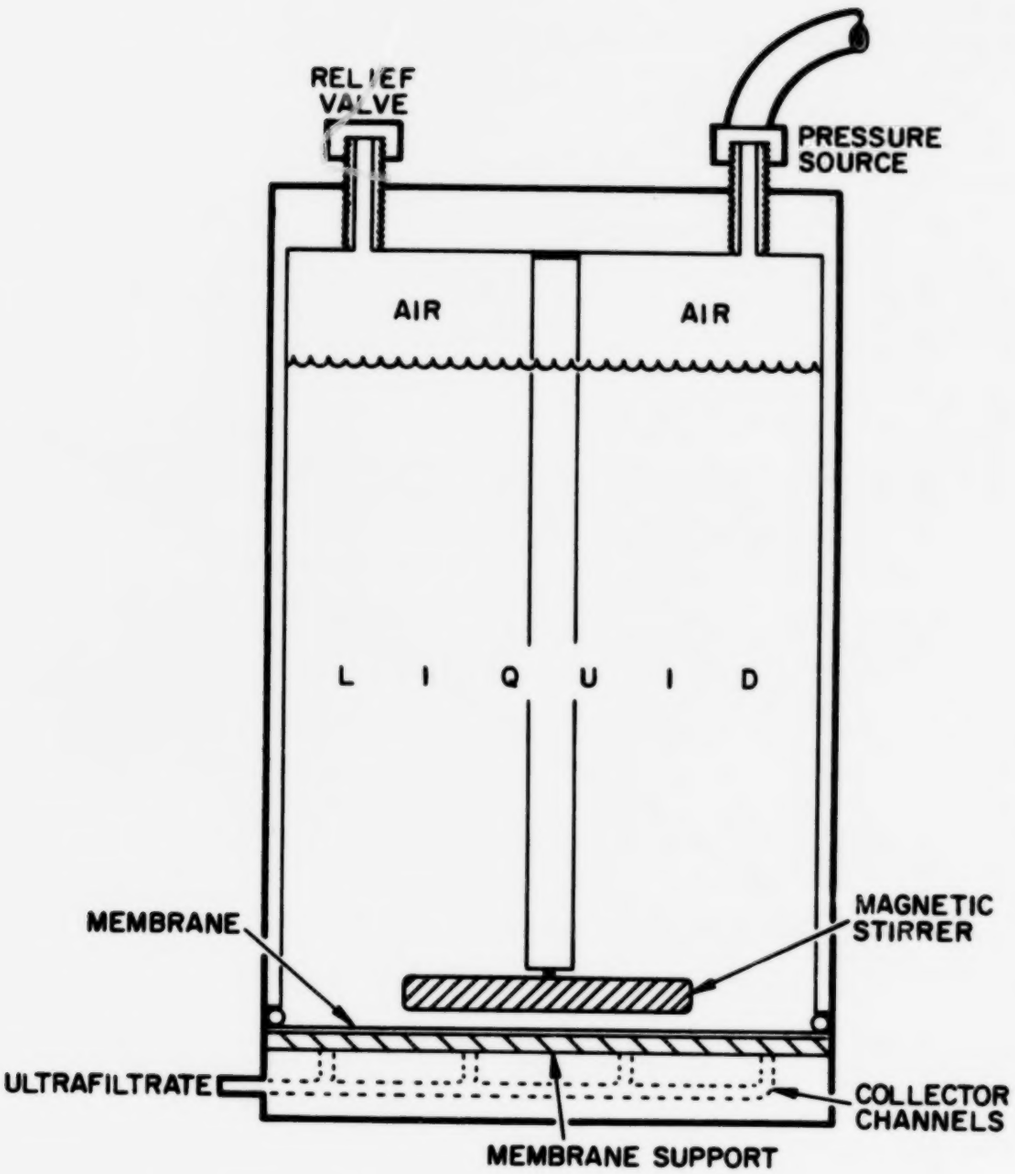
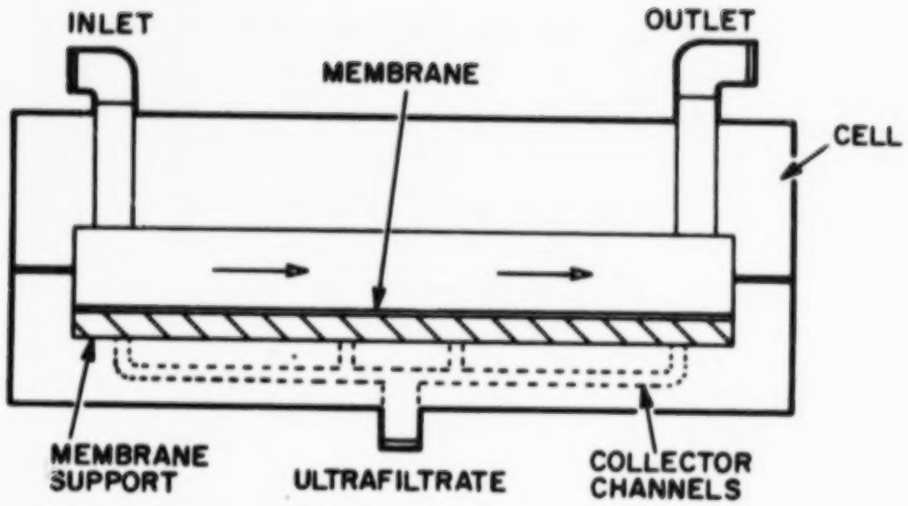


FIGURE 4. MODEL 400 CELL



INTERNAL DIMENSIONS
4.0 in. x 1.5 in. x 0.125 in.
(VERTICAL SCALE EXAGGERATED)

FIGURE 5. SHORT PATH THIN CHANNEL CELL

Table II

Laboratory Flocculation of High COD Brockton Secondary Effluent

Secondary Effluent COD, mg/l	Aqua Nuchar A mg/l	Primafloc C-7 mg/l	Settling Time	Settled Volume ml	% Solids	% Solids Recovery in Sediment
240	500	3	8-10 hrs	34	6.6	87
240	500	5		36	6.2	88
240	500	7		42	5.4	89
240	500	9	20 min	48	4.8	90
240	600	3	8-10 hrs	38	6.5	86
240	700	3	8-10 hrs	43	6.4	85

Settling trials were carried out in 600 ml beakers containing 400 ml of effluent.

Settled volumes were measured after 48 hrs. of quiescent standing from the time of C-7 addition.

support and collector channels leading to the ultrafiltrate port. The short channel cell is particularly well suited for continuous dewatering of rapidly ultrafilterable materials.

The Long Path Thin Channel Cell

An Amicon Model TC-1 thin channel cell was used for most of the dewatering runs in this program. An exploded view of this cell is shown in Figure 6 and the principal of operation is shown schematically in Figure 7. The number, length and thickness of the channels are determined by the spacer plate shown in Figure 6.

Most of the runs in this program were carried out using a spacer plate with two parallel spiral channels each of which was 28.8 in. long, 1/4 in. wide and 0.060 in. deep. In general the feed stream enters the two ports near the periphery of the spacer. The two parallel spiral streams join just before exiting through the center port. Note that only one channel is shown in the schematic Figure 7.

As in the short channel cell, the channel itself is bounded on one side by the membrane which is supported by a porous sintered polyethylene disc. Liquid passes through the membrane and the disc into collecting channels and out the ultrafiltrate port.

The effective membrane area of the two parallel channels is (2)(28.8 in.)(0.25 in.) = 14.4 $in.^2$ = 0.10 ft^2.

Under typical operating conditions, a high flow rate along the channel is maintained in order to minimize concentration polarization, i.e. the accumulation of retained solids against the membrane's surface which causes reduction in flux. A high, but not unusual, circulation rate of 1 gpm through two 0.060 in. x 0.25 in. channels corresponds to a linear velocity of 10.5 ft/sec.

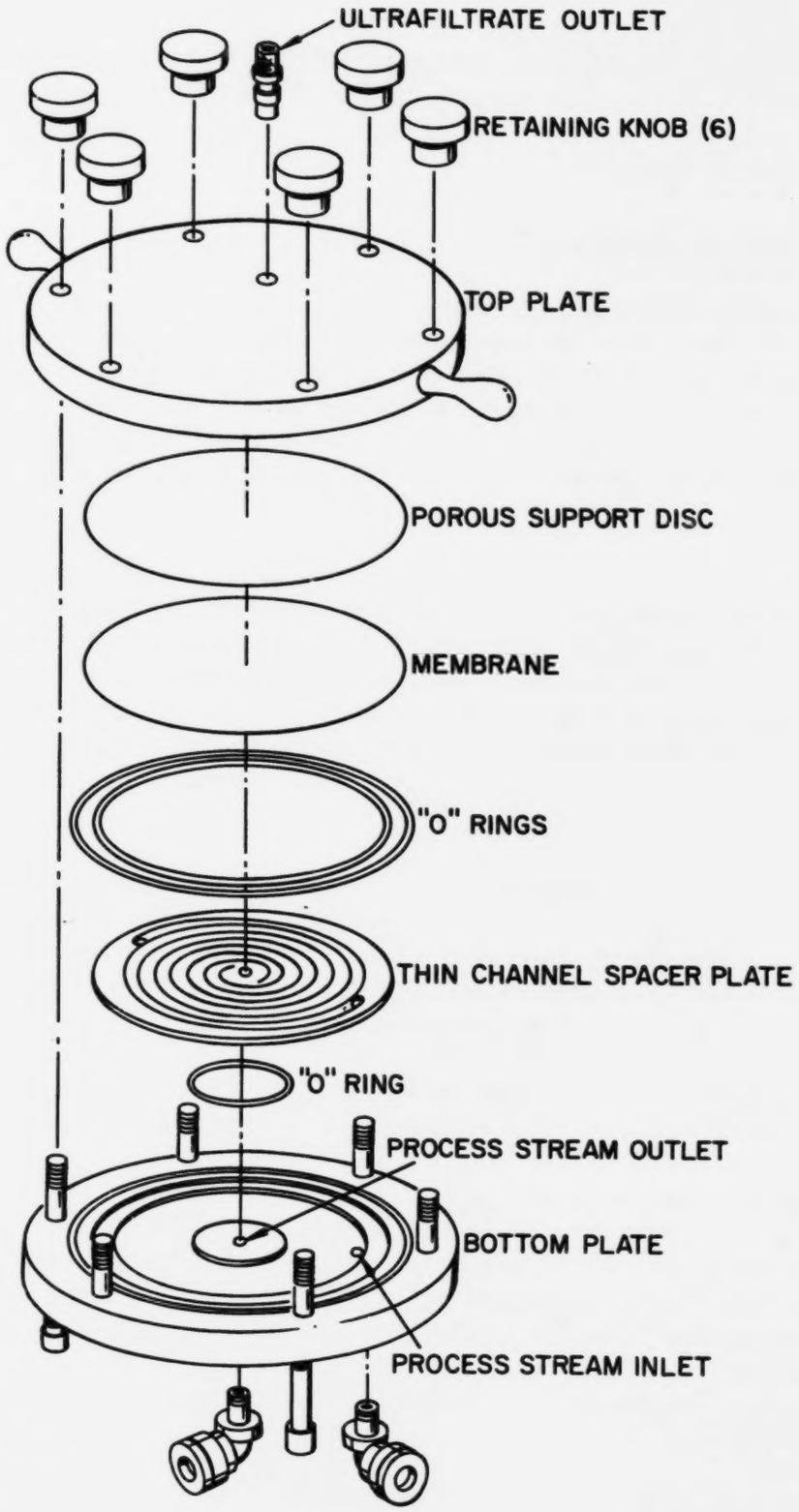


FIGURE 6. EXPLODED VIEW OF AMICON TC-1 CELL

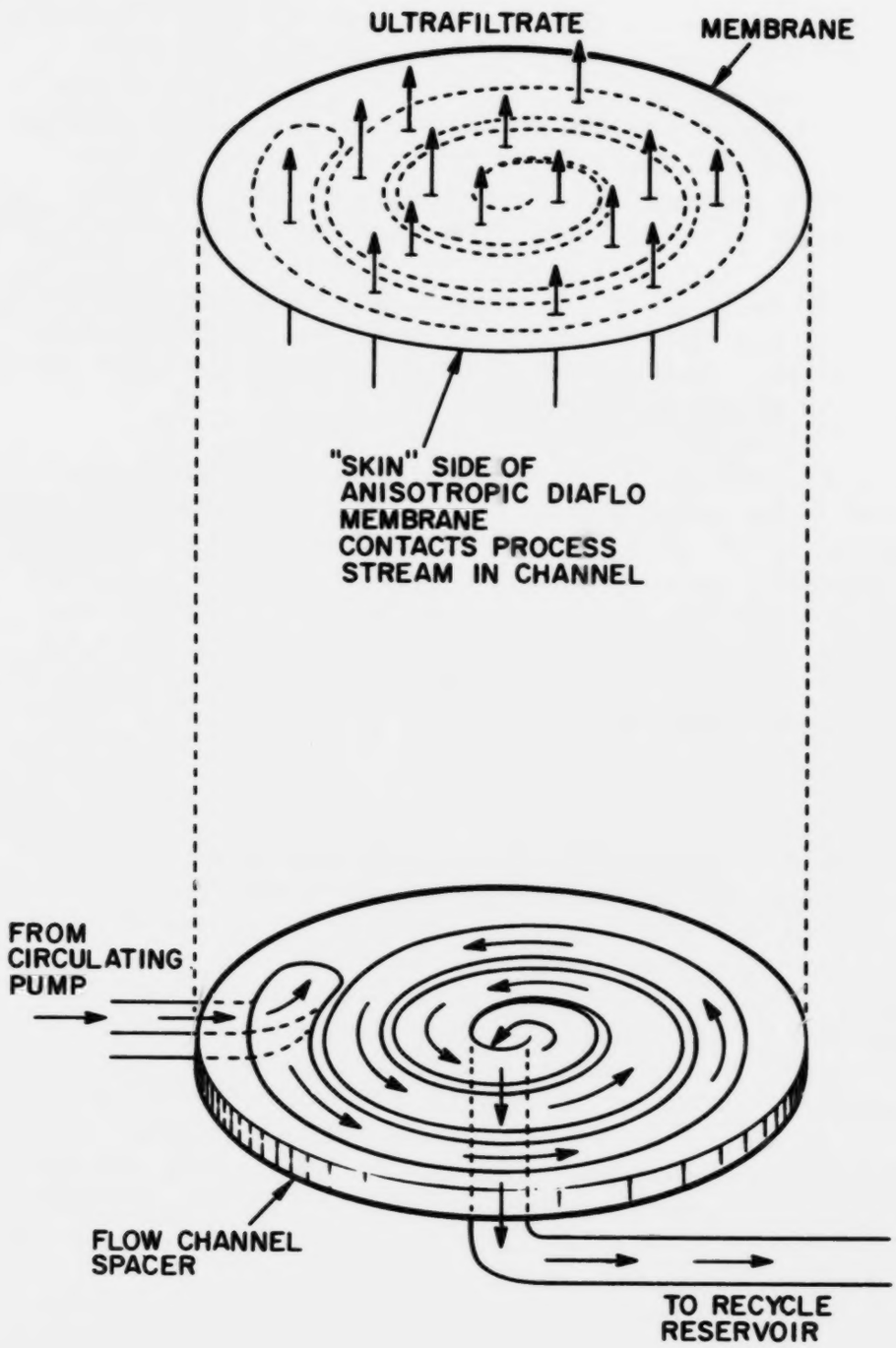


FIGURE 7. LIQUID FLOW PATTERN IN TC-1 MEMBRANE MODULE

The circulation rate through the channel far exceeds the amount of liquid passing through the membrane. Under typical operating conditions 1/2 to 1% of the circulating feed stream is dewatered per pass through the cell. Thus, the volume of liquid entering and leaving the upstream side of the cell is typically 50-100 times greater than that leaving as ultrafiltrate.

The apparatus used to circulate the feed, and monitor flow rates and pressure drops is shown schematically in Figure 10. The actual apparatus used for Runs 8B-1 to 9B-3 is shown in Figure 9.

After the completion of Run 9B-3, it was recognized that the gear pump (which is part of the standard apparatus shown in Figure 9) had inadequate pumping power as well as poor wearing qualities for the high concentration of abrasive slurries which were being produced. A Moyno worm drive pump was used for the remainder of the runs. The hardware system used with the Moyno is shown schematically in Figure 10.

Membranes

Inasmuch as the particle size of the material to be retained, Aqua Nuchar A, has a median size of 11μ, ultrafiltration media with relatively large pores could be employed. The particular medium used for most of the dewatering runs in this program was an Amicon XM-100 membrane which has a nominal pore diameter of about 0.01 microns. A cross section of a typical ultrafiltration membrane is shown in Figure 11. Note that the structure varies from a very dense "skin" side to a more highly porous spongy side. The skin side, which has the 100 Å diameter pores (below the limit of resolution of the photograph) is placed in contact with the feed stream. No carbon particles in excess of 0.01 micron diameter or macromolecular material of greater than 250,000 molecular weight can penetrate the membrane skin. The XM-100 membrane has a nominal distilled water flux rate of 200-250 gfd at 10 psi transmembrane pressure.

In several preliminary runs, a spun bonded polyolefin sheet (Tyvek, E.I. DuPont de Nemours and Co., Inc.) was used as the filtration medium.

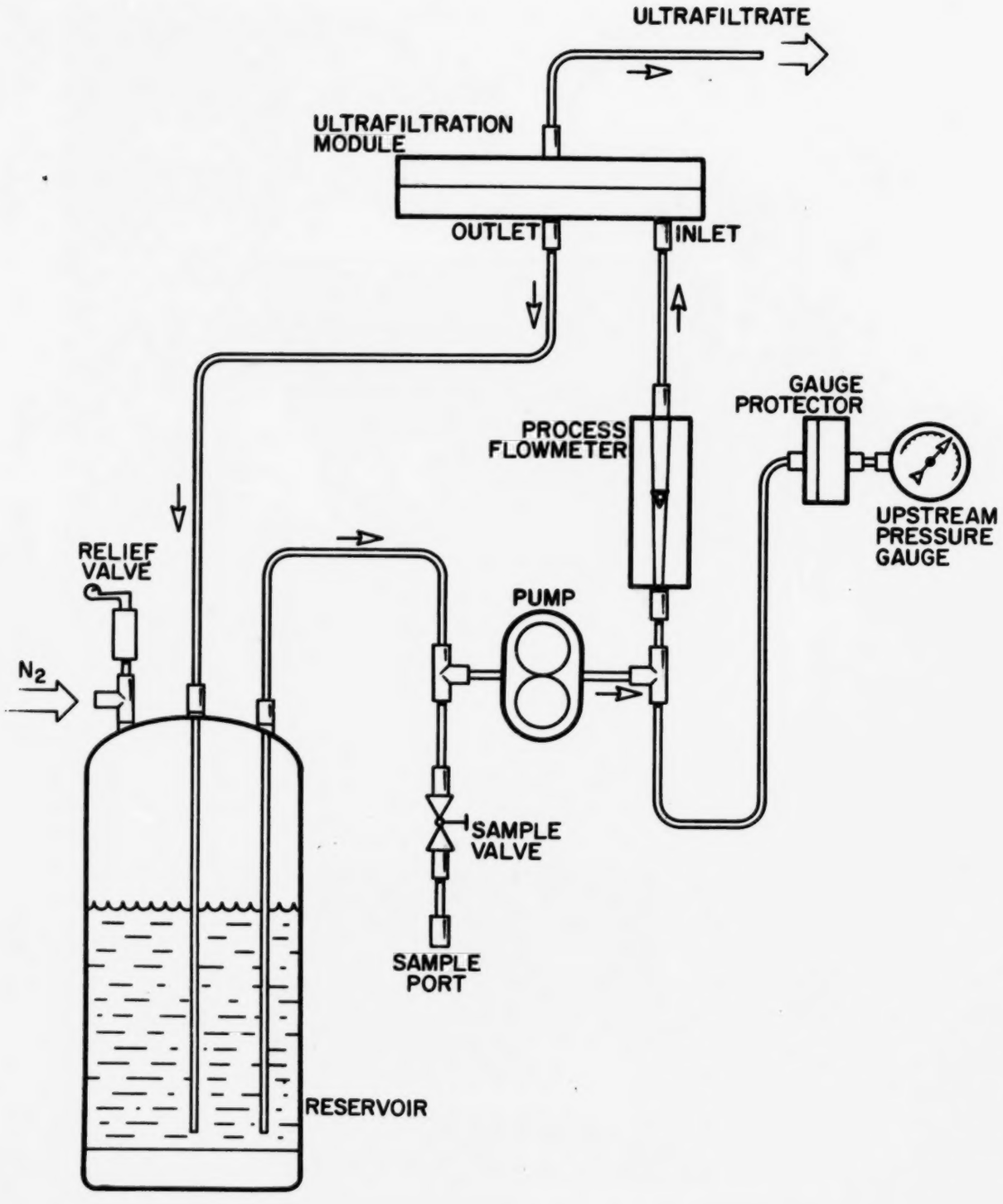


FIGURE 8. TC-1 ULTRAFILTRATION SYSTEM SCHEMATIC

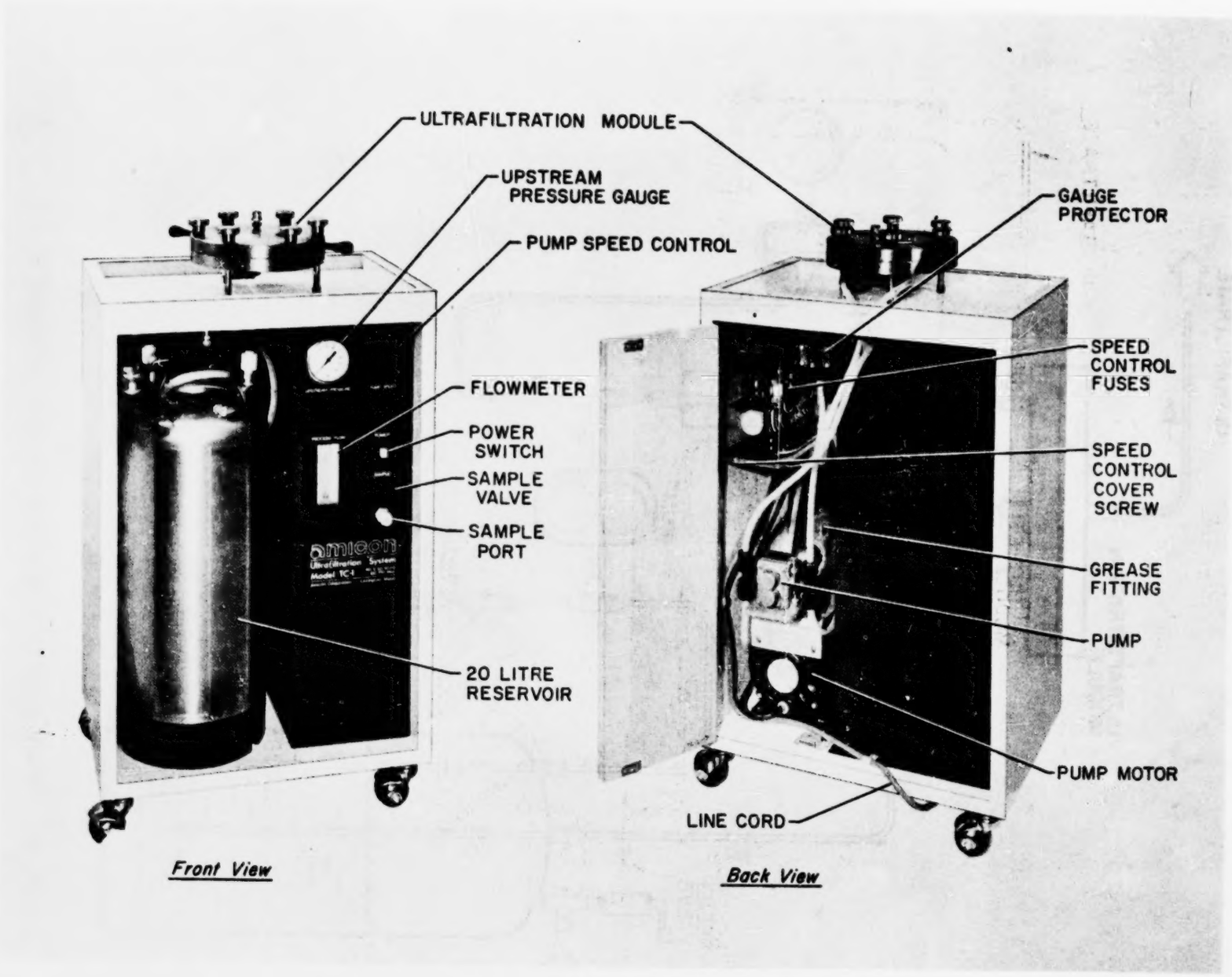


FIGURE 9. TC-1 ULTRAFILTRATION SYSTEM

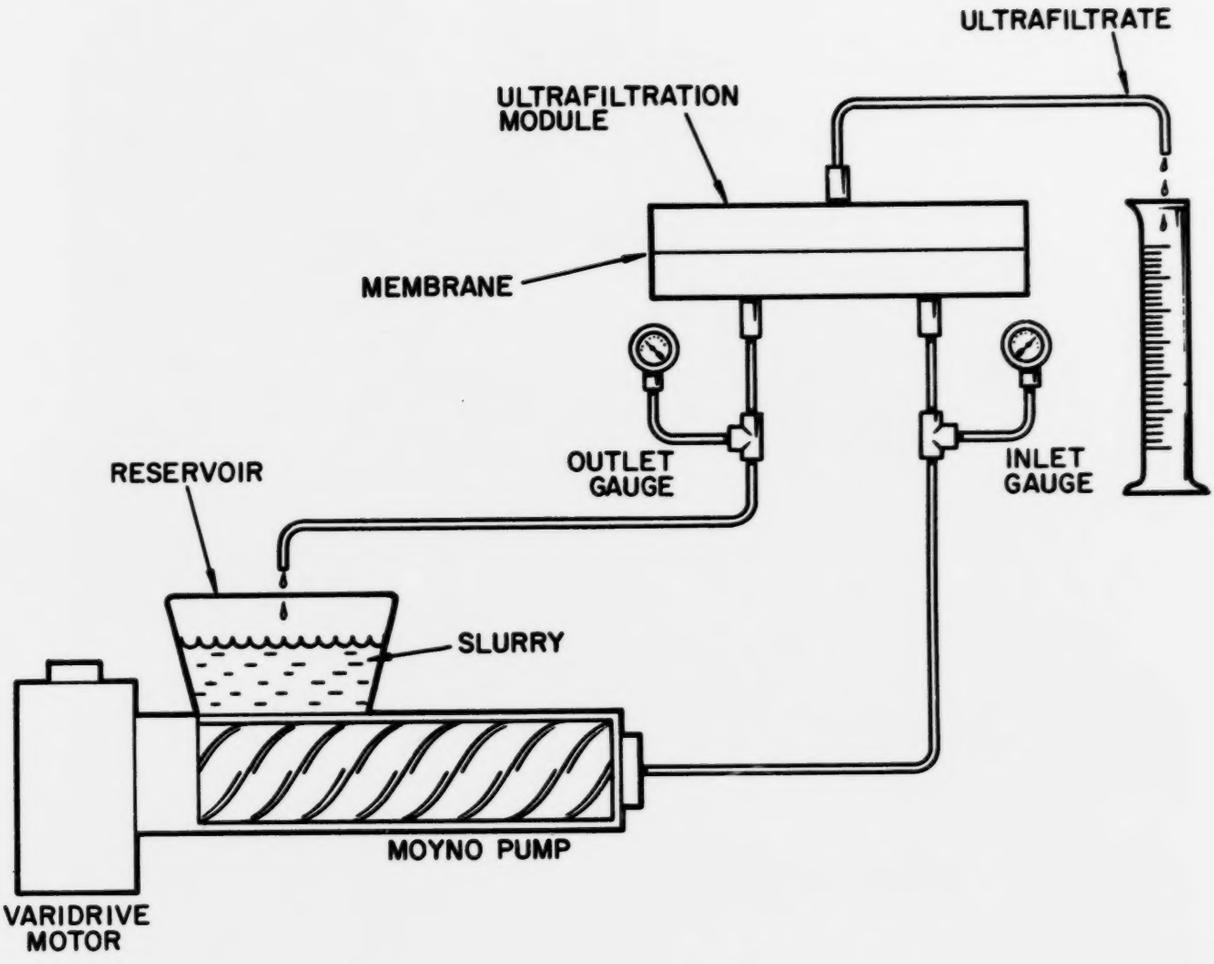


FIGURE 10. TC-1 MOYNO ULTRAFILTRATION SYSTEM

FIGURE 11. CROSS SECTION OF SURFACE OF XM-100 MEMBRANE

NOT REPRODUCIBLE

DEWATERING RUNS

Virgin Carbon

Initial dewatering runs were carried out with virgin carbon slurries in the TC-1 unit (See Figure 9). Runs were of short duration, terminated by carbon plugging the channel. Subsequent experiments demonstrated that the plugging was due to the high efficiency of dewatering in this unit. The combination of high membrane flux and the highly efficient thin channel device in conjunction with the easily dewatered virgin carbon gave rise to these plugging problems. The results of the dewatering runs on virgin carbon are summarized in Table III. It was concluded from these runs that if it were possible to limit the dewatering of the carbon to 20% solids in a single pass, then runs of longer duration could be made in which the entire contents of the reservoir were concentrated.

In an effort to determine independently the dewatering rate and ultimate carbon solids achievable with virgin carbon, a 10% Aqua Nuchar A slurry was dewatered in the batch cell under conditions of no recirculation or stirring. The resultant cake assayed 31% solids.

Inspection of the cake suggested that this 31% solids level was a true upper limit and not an artifact due to some thixotropic behavior caused by weak structuring of the carbon. Thus it is likely that any mechanical means of dewatering the carbon - ultrafiltration, centrifugation, etc. - can at best only approach this 31% level as an upper limit.

The average water flux rate through the membrane during this dewatering was about 80 gfd at 2 psi transmembrane pressure. Under identical conditions distilled water fluxes at the rate of 150 gfd. The lower flux rate of the carbon slurry indicates that the hydraulic permeability of the carbon filter cake rather than the permeability of the membrane partially controls flux rate.

Table III

Dewatering of Virgin Carbon in the TC-1 Cell-Gear Pump

XM-100 membrane Recirculation rate: 0.132 gpm (estimated)
Feed: 1.0 gal of 10% Aqua Nuchar A
Membrane area = 0.1 ft^2

Pressure* (psi) Entering/ Leaving	Duration of Run (min)	Ultrafiltrate	Run Terminated by	Average Flux Rate (gfd)	% Solids in Plug	Comments
42/10	15	0.132 gal clear	single plug in channel	127	not determined	30 mil channel 2 paths 30" long
20/0	11	0.125 gal clear	"	164	25%	"
20/0	12	0.094 gal clear	"	113	20%	"
20/0	4	0.021 gal clear	"	75	21%	inlet holes in channel doubled in size
60/0	3	0.015 gal clear	plug filling entire channel	72	28%	60 mil channel 4 paths 18" long

* Pressure on the ultrafiltrate side of the membrane is always zero. The "average" transmembrane pressure is usually taken as the mean of the inlet and outlet pressures, e.g., 42/10 psi "averages" to 26 psi.

Thus it would be of little value to use a membrane of higher distilled water flux rate. Subsequent experiments, using more open media than the XM-100 membrane, corroborate this.

Comparison of Dewatering Cells Using Lebanon Samples

Two differing lots of spent carbon, Lebanon Nos. 2 and 3 were used in the following runs in which the dewatering efficiencies of the three types of ultrafiltration cells were compared. Lebanon No. 2 was completely free of odor, similar in appearance and dewatering characteristics to a virgin carbon slurry. In contrast, Lebanon No. 3 was foul smelling, contained numerous microorganisms (visible under the microscope) and was comparatively difficult to dewater. In retrospect, it is quite likely that the Lebanon No. 2 was a thermally regenerated carbon.

Table IV shows the results of a series of dewatering runs made in the batch ultrafiltration cell. Lebanon No. 2 was easily dewatered at a high flux rate using either an XM-100 membrane, a piece of Tyvek spun bonded polyolefin, or a piece of Whatman No. 5 filter paper. The filtrate was quite clear in all cases. A magnetic stirrer was used in each case which stalled when the solids content reached about 14%. In each case with Lebanon No. 2 the slurry dewatered to the same 30% solids dry cake. The higher fluxes using the Tyvek and filter paper are due to the higher pressures employed.

In contrast, Lebanon No. 3 dewatered with much more difficulty in the batch cell. In spite of the higher pressure employed its flux was only 20 gfd instead of the 110 gfd found with Lebanon No. 2. The maximum solids attainable in the batch cell was only 19% in contrast to the 30% attainable with Lebanon No. 2 and virgin carbon.

Dewatering in the Short Channel Cell

Table V shows the results of dewatering experiments in the short channel cell. About one gallon of feed was recirculated through this cell using the equipment shown in Figure 9 with the replacement of the short channel cell for the TC-1 shown in the figure. Using an XM-100 membrane,

TABLE IV

Dewatering Runs in the Model 400 Batch Ultrafiltration Cell

Volume of charge: 0.08 gallons
All filtrates were clear.
All runs were magnetically stirred.

Charge	% Solids Initial	% Solids Final	Transmembrane Pressure	Average Flux	Filter Medium
Lebanon No. 2	9	ca 30*	3 psi	110 gfd	XM-100
No. 2	9	ca 30*	3 initially 30 psi final	350 gfd	Tyvek
No. 2	9	ca 30*	20 psi	475 gfd	Whatman No. 5
No. 3	11	19	25 psi	20 gfd	XM-100

*Stirrer stopped at 14% solids.

TABLE V

Dewatering Runs in the Short Channel Cell-Gear Pump

XM-100 membrane, circulation rate ~0.132 gpm (estimated)

Charge	Volume Circulated (gallons)	% Solids Initial	% Solids Final	Pressure Entering/ Leaving (psi)	Flux	Comments
Lebanon No. 2	0.950	9	18	6/0 initial 8/0 final	90 gfd	clear filtrate, 6 hr run
No. 3	0.950	11	11.5	10/0	26 gfd	1/16 in. slime buildup on membrane - 0.045 gal. of ultrafiltrate in 1 hr
No.3	0.950	11	12	20/0 initial 75/0 final	30 gfd	same 1/16 in. gel layer - 0.1 gal. of ultrafiltrate in 1 hr

the Lebanon No. 2 slurry (regenerated carbon) was dewatered from 9 to 18% solids at the low transmembrane pressure of 6-8 psi, at a 90 gfd flux rate. Although the flux rate was comparable to that obtained with virgin carbon slurries, no plugging was observed in this short channel cell.

In contrast, the dewatering of Lebanon No. 3 was much more difficult. After one hour the feed had dewatered from 11 to only 11.5% solids at a flux rate of 26 gfd. The flux then dropped to a very low value at which time the run was discontinued. Upon disassembling the cell, a 1/16 in. layer of slime or gel (which occupied half the thickness of the 1/8 in. channel) was observed on the membrane - in all likelihood causing the reduced flux. In an additional run with Lebanon No. 3 at higher pressure (25 psi instead of 10) the flux rate increased by only 15%.

A flux rate that is only weakly dependent on pressure is characteristic of ultrafiltration conditions in the gel controlled region, a clearly undesirable operating condition.

Dewatering in the Thin Channel Cell

The results of dewatering experiments in the spiral module TC-1 is shown in Table VI. Using Lebanon No. 2, the run terminated with a plugged channel after several minutes of operation, as did virgin carbon in this cell.

Relative to the short channel cell, the TC-1 cell is far more efficient due to its longer channel length and smaller channel height. The 60 psi pressure at the cell inlet resulted from channel plugging.

In contrast to Lebanon No. 2, the Lebanon No. 3 was more successfully dewatered in the thin channel unit. Since the intrinsic flux rate of this material is lower than that of Lebanon No. 2, no plugging was observed as the slurry was concentrated from 11% to 19% solids. The pressure rose gradually from 10 to 20 psi during the course of the run. The 70 gfd flux is the highest observed in any of the cells for Lebanon No. 3. Very little slime buildup was noticed on the membrane.

TABLE VI

Dewatering Runs in the Spiral Module TC-1-Gear Pump

XM-100 membrane, circulation rate ~0.132 gpm (estimated)
Linear velocity: 1.41 ft/sec
Channel length: 16 in. (each one)
Channel depth: 0.060 in.

Charge	Volume Circulated (gallons)	% Solids Initial	% Solids Final	Pressure Entering/ Leaving (psi)	Flux	Comments
Lebanon No. 2	0.925	9	9.5	5/0 initial 60/0 final	low (less than 20 gfd)	plugged after passage of 0.015 gallons of ultra-filtrate
No. 3	0.925	11	19	10/0 initial 20/0 final		

The dewatering properties of Lebanon No.3 are probably more typical of those of spent carbon slurries. For such systems, the long thin channel device is most suitable. The high shear conditions in the thin channel keep the membrane surface clean, thus inhibiting the accumulation of solids. For such easily dewatered materials, however, as Lebanon No. 2, highly efficient dewatering devices can lead to plugging.

Any full scale ultrafiltrative dewatering device should be able to accommodate both types of sludge and should therefore be equipped with suitable control devices to reduce the transmembrane pressure when easily dewaterable sludges are pumped through the unit. (See later discussion.)

Thin Channel Runs Using Brockton Samples With the Gear Pump

Inasmuch as the previous runs demonstrated that the most efficient dewatering device for spent carbon slurries was the thin channel spiral module, a second series of experiments was undertaken to optimize the dewatering procedure using spent carbon from Brockton. The first of these runs was carried out using Tyvek as the filter medium, in order to determine whether a microporous ultrafiltration membrane was in fact necessary in view of the relatively large size of the carbon particles to be removed. A spent carbon slurry from Brockton (See Table VII) was circulated through the system using both 0.060 in. and 0.030 in. thick channels. Both runs exhibited rather low flux rates especially considering the low carbon concentration at which dewatering took place. The run with the 0.030 in. channel was terminated by plugging.

The low flux and tendency to plug were quite likely due to the very high COD of the secondary effluent used to prepare the carbon slurry; 410 mg/l of COD adsorbed by 200 mg/l of activated carbon tends to result in a noticeably slimy slurry. Since the distilled water flux for Tyvek at these transmembrane pressures is several hundred gfd, the low fluxes observed are a strong indication of gel layer formation.

A subsequent batch of spent carbon prepared at Brockton, 8C, was derived from a secondary effluent much lower in COD, 75 mg/l. Improved fluxes of 41 and 50 gfd were observed at lower transmembrane pressures. These fluxes are comparable to the 70 gfd found for Lebanon No. 3 slurry shown in Table VI.

For the runs in Table VII, the circulation rate was about 0.32 to 0.37 gpm. Since the initial volume of spent carbon slurry charged to the reservoir is about 0.80 gallons, the time for complete turnover, assuming good mixing of the reservoir contents (not always the case), during the initial portion of each run is about 2-2.5 minutes. During the latter portion of these runs, when the slurry has been concentrated about fourfold, the turnover time is about once every half minute. The rate of ultrafiltration during the major portion of these runs is such that approximately 1% of the total water is removed per minute, or 0.5% per pass.

Inspection of the equipment after the completion of Runs 8C-1 and 8C-2 suggested that the inability to achieve solids contents higher than about 16 or 17% was due to the inability of the gear pump to suck up the locally concentrated (due to lack of stirring) slurry from the reservoir.

Consequently during the series of runs beginning with 9B-1, a stirrer was placed in the reservoir during the latter, high viscosity portion of each run. As can be seen from Table VIII, this procedure permitted higher final solids levels to be obtained.

The 9B series of runs were also carried out under slightly different conditions during the early portion of each run, while the solids concentration was increasing up to 10% with very little viscosity change. During this part of the run, the circulation loop was sealed from the atmosphere so that the system could be pressurized by applying 15 psi nitrogen to the reservoir. At a system pressure of 15 psi, if the head pressure of the pump is 10 psi (a function of the circulation rate) the actual inlet pressure to the cell is 10 + 15 = 25 psi. Since the pressure head loss takes place almost totally within the cell,

TABLE VII

Dewatering Runs in the Spiral Module TC-1 Cell-Gear Pump

Circulation rate: 0.32-0.37 gallons/minute
Linear velocity: 3.7 ft/sec for 60 mil channel

Charge	COD of Sec. Effluent Source mg/l	Volume Circulated (gal)	% Solids Initial	% Solids Final	Pressure Entering/ Leaving (psi)	Flux	Filter	Comments
Brockton 8B-1	410	0.80	3.8	14.6	20/0	26 gfd	Tyvek	60 mil channel; gel buildup
8B-2	410	0.80	3.8	11.0	20/0	16 gfd	Tyvek	30 mil channel; plugged
8C-1	75	0.80	4.0	16.7	between 7/0 and 10/0 psi up to 10% solids	41 gfd	XM-100	60 mil channel
8C-2	75	0.77	4.0	16.0	between 7/0 and 10/0 psi up to 10% solids between 10/0 and 22/0 psi up to 16% solids	50 gfd	XM-100	60 mil channel

TABLE VIII

Dewatering Runs in the Spiral Module TC-1 Cell-Gear Pump

XM-100 membrane Circulated rate: 0.40 gpm (estimated)
Linear velocity: 4.3 ft/sec
Channel thickness: 0.060 inches
Channel length: 29 inches

Charge	COD of Sec. Effluent Source mg/l	Volume Circulated (gallons)	% Solids Initial	% Solids Final	Transmembrane Pressure Entering/ Leaving (psi)	Flux (gfd)	Comments
Brockton			Step 1				
9B-1	75*	0.99	4.5	9.7	25/15	65	ultrafiltrate COD 10 mg/l
			Step 2				
			9.7	24.5	25/0	50	
			Step 1				
9B-2	75	1.02	4.0	9.0	25/15	45	
			Step 2				
			9.0	22.0	25/0	55	
9B-3	65	1.85	5.0	17.5	30/0	35	used for viscosity and dispersant studies

*The COD of the ultrafiltrate of Run 9B-1 was 10 mg/l.

the outlet pressure is 0 + 15 = 15 psi. The pressure on the downstream or ultrafiltrate side of the membrane is always zero (atmospheric). Thus, in the experiment above, inlet pressure of 25 psi and an outlet pressure of 15 psi give an average transmembrane pressure of 20 psi.

Thin Channel Runs With the Moyno Pump

Due to wear and inadequate capacity for pumping carbon slurries a substitute for the gear pump was sought. Based on the experience of the FWPCA in pumping carbon slurries, the gear pump was replaced by a Moyno Pump. The pumping arrangement was somewhat different with the Moyno as shown in Figure 10. Note in particular that the separate reservoir was eliminated; rather the exiting feed stream returning from the ultrafiltration cell was discharged directly into a hopper built directly over the feed entrance of the Moyno.

The initial runs with the Moyno pump were carried out with virgin carbon slurries. The results are shown as runs 9-4 and 9-5 in Table IX. It was immediately apparent that operations with the Moyno pump substituted for the gear pump were very much improved. A circulation rate of 0.92 pgm (over twice the maximum achievable with the gear pump) was easily achieved which resulted in fluxes of 200 and 250 gfd. Compare this data with the virgin carbon run data using the gear pump in Table III where the fluxes were 75-150 gfd.

The first run with the Moyno pump on a spent carbon slurry was performed on material prepared at Brockton, designated 9-6. In order to achieve the high dewatering level of 24.8%, it was necessary to increase the speed of the Moyno pump as the run progressed. Increasing the speed increased the pressure drop through the cell (along the channel). In Run 9-6 this pressure drop increased from 10 psi at the beginning of the run to 80 psi by the end of the run. A high flux rate was maintained throughout most of the run, averaging 125 gfd, falling off somewhat as the solids increased above the 20% level.

TABLE IX

Dewatering Runs in the Spiral Module TC-1 Cell-Moyno Pump

XM-100 membrane Circulation Rate: 0.80 gpm: Moyno Pump
Fluid velocity: 8.5 ft/sec
Channel depth: 0.060 in. Channel length: 29 in.

Charge	COD of Sec. Effluent Source mg/l	Volume Circulated (gallons)	% Solids Initial	% Solids Final	Pressure Entering/Leaving psi Initial	Pressure Entering/Leaving psi Final	Flux (gfd)	Comments
Virgin carbon 9-4	none	0.92	10.0	19.2	unknown		200	
Virgin carbon 9-5	none	0.92	10.0	20.0	15/0	30/0	250	
Brockton 10-1	80	1.98	5.8	24.8	10/0	80/0	125	Temp. rose to 42°C Final flux at 125 gfd.
Brockton 10-2	65	1.85	4.1	24.0	20/0	40/0	140	COD of filtrate 56 mg/l. Temp. rose to 50°C. Final flux at 100 gfd.
Lebanon 5 10-3	unknown	1.50	10.2	31.0	20/0	80/0	70	Temp. rose to 65°C Ultrafiltrate turned pale green prior to run term.
Lebanon 5 10-4	unknown	0.92	10.2	17.0	20/0	35/0	70	
Brockton 10-5	114/76*	----------See Tables----------						Exten. running to meas. flux, pres., viscosity parameters.

* Total COD is 114 mg/l; after filtration, the COD is 76 mg/l, the difference representing the COD of the suspended solids.

The behavior of the carbon slurry on Run 10-1 was similar. The temperature was monitored during this run. Starting at room temperature, it began climbing during the latter portion of the run (when the pump speed was increased), resulting in a final temperature of 42°C. The increased temperature decreased the viscosity of the slurry as was observed on subsequent runs. Note that even at the high final solids level of 24% the flux rate was maintained at 125 gfd.

Run 10-2 was similar to Run 10-1 except that a greater volume was circulated. Thus the longer duration of the run resulted in a greater temperature rise which in turn permitted an extra 2% final dewatering.

Run 10-3 was performed on Lebanon No. 5. The material as received had a characteristic anaerobic sulfide odor not observable in the freshly prepared Brockton slurries. Lebanon No. 5 dewatered to an unusually high 31.0% solids. Inasmuch as this high a solids level was not approached with any other carbon slurry, it is suspected that this sample contained an unusually high amount of organic matter, possibly contained within the pores of the carbon particles. The presence of organic matter would also be consistent with the lower flux values of 70 gfd that were found for this slurry. By the time this run was terminated the temperature of the slurry had risen to 65°C from room temperature; in addition the final half gallon of ultrafiltrate had a pale green cast.

The same slurry was used in Run 10-4, but the dewatering was terminated purposely after the solids concentration had reached 17% in order to prepare a material for testing the effects of dispersing agents on viscosity. The viscosity experiments are reported in the section on Rheological Studies.

Long Term Dewatering Runs

Having found the general operating conditions needed to obtain high rates of dewatering and high final solids concentrations, two long term dewatering runs were carried out. The purpose was to obtain more quantitative relationships between operating conditions and dewatering

rates and in particular to determine whether membrane performance deteriorates with time. As will be shown later in the Economics, the feasibility of membrane dewatering is most highly dependent on membrane lifetime and average flux throughout this lifetime.

The two long term dewatering runs were carried out for 6 and 9 days using spent carbon slurries prepared at Brockton. As in all the previous experiments, no carbon was found in the ultrafiltrate during these runs. Long term flux rates of about 45 gfd were maintained at room temperature operation (ca 27°C). The cell was immersed in a cooling bath for this portion of the experiment. At somewhat elevated temperatures (42°C), fluxes of the order of 65 gfd were obtained, but no sustained runs at 42°C were carried out. Although membrane flux rates do appear to decrease slowly with time, no evidence of plugging or gross destruction of the XM-100 membrane was observed. Some wear was observed in Run 11-1 near the entrance and exit ports. The COD's of the ultrafiltrates were typically in the range of 25-35 mg/l, indicating no breakthrough. Details of these runs follow.

A long term run designated 11-1 was made as follows:

A 5.6% solids spent carbon slurry from Brockton was prepared in the usual manner. An initial charge of 2.35 gal. was dewatered to a 10.0% solids slurry in a period of about 3 hours at an average flux rate of about 100 gfd. A slight temperature increase from room temperature to 27°C was observed. The long term dewatering run was considered to commence with the resultant 1.32 gallons of 10% solids slurry. This material was recirculated through the 0.060 in. deep channel TC-1 cell using the Moyno pump for 144 hours. The membrane was an XM-100. The ultrafiltrate was recombined with the circulating slurry in the reservoir above the Moyno pump hopper. During the course of the run evaporative losses which took place from the reservoir surface increased the solids level to 14% by the end of the run. In order to maintain isothermal operating conditions during most of the run, the cell was immersed in a water bath. When flux rates at the elevated temperature conditions, resulting from the pumping work, were to be determined, the cell was removed

from the water bath (during the last 6 hours of the run). The course of the run is shown in some detail in Figure 12.

During the first 24 hours of the run the circulation velocity was maintained at the relatively low level of 5.1 ft/sec which resulted in an average transmembrane pressure of 7 psi. Over the next few hours in a series of two steps the circulation velocity was increased to 8.5 ft/sec causing the pressure at the cell entrance to climb to 18 psi. Note however that the rise in transmembrane pressure from 7 to 9 psi caused more than a proportional increase in the flux rate of from about 20 to 40 gfd.

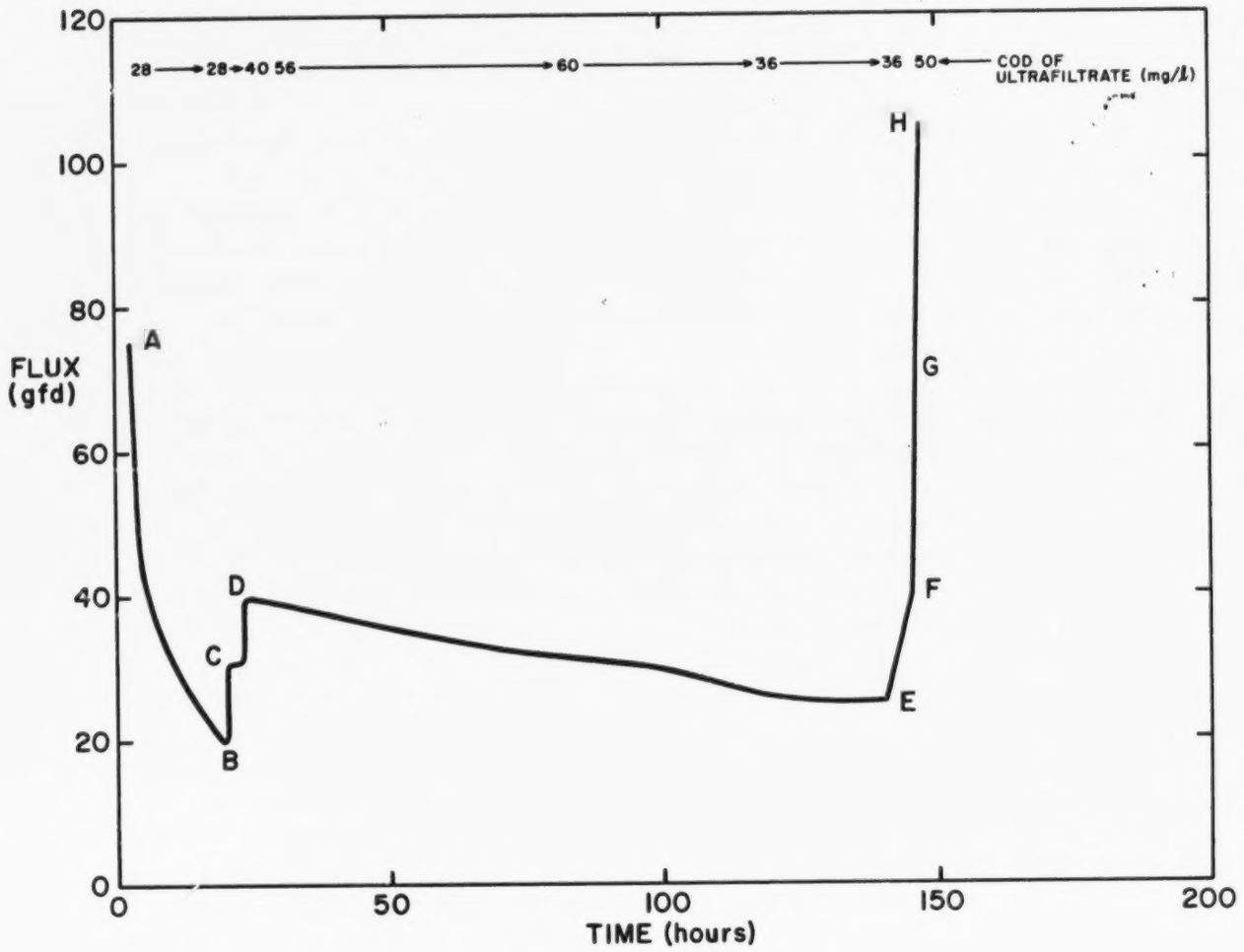
During the next 120 hours no changes in operating conditions were made. During this time the flux rate decreased from 40 to 25 gfd. The extent to which this decrease in flux is due to membrane compaction or is due to the slowly increasing solids contents of the slurry from evaporation is not known.

At the 142 hour mark, the cooling was removed resulting in a temperature rise from 25 to 42°C over the next 4 hours. The transmembrane pressure increased, but the flux rate increased more than proportionally.

The circulation velocity was then increased to 11.3 ft/sec and resulted in an average transmembrane pressure of 17 psi and the rather high flux value of 110 gfd.

The run was then terminated and the cell was disassembled for inspection of the membrane. The membrane directly in the channel areas appeared to be essentially unattacked. Some erosion had taken place near the exit region; in this area it appeared that some small carbon granules had penetrated into the subskin region of the membrane.

During the entire course of the run the ultrafiltrate samples were clear. COD determinations were also made on the ultrafiltrate from time to time in order to have advance warning of impending membrane failure. The COD values are located along the top of Figure 13. These values appear to show no consistent rise or decline with time.



TC-1 CELL
MOYNO PUMP
XM-100 MEMBRANE
CHANNEL HEIGHT 0.060 in.
10% SOLIDS/5 liters

RUN STARTED AT 2.75 hours MEMBRANE LIFE

A $\Delta P = 7$ psi V=5.1 ft/sec
B $\Delta P = 6$ psi V=5.1 ft/sec
C $\Delta P = 7.5$ psi V=6.8 ft/sec
D $\Delta P = 9$ psi V=8.5 ft/sec
E COOLING REMOVED, $\Delta P = 9$ psi T=25°C
F $\Delta P = 11$ psi V=11.3 ft/sec T=42°C
G $\Delta P = 14$ psi V=11.3 ft/sec
H $\Delta P = 17$ psi V=11.3 ft/sec T=48°C

FIGURE 12. LONG TERM DEWATERING RUN 11-1

A second run designated 11-2 was then made as follows:

As in the previous run the spent carbon slurry was prepared at Brockton in the usual way. The COD of the secondary effluent from which the slurry was prepared was 100 mg/l of which 42 mg/l was removable by sand filtration. The COD of the clear supernatant after the carbon flocculation was 20 mg/l. Details of the run are shown in Figure 13.

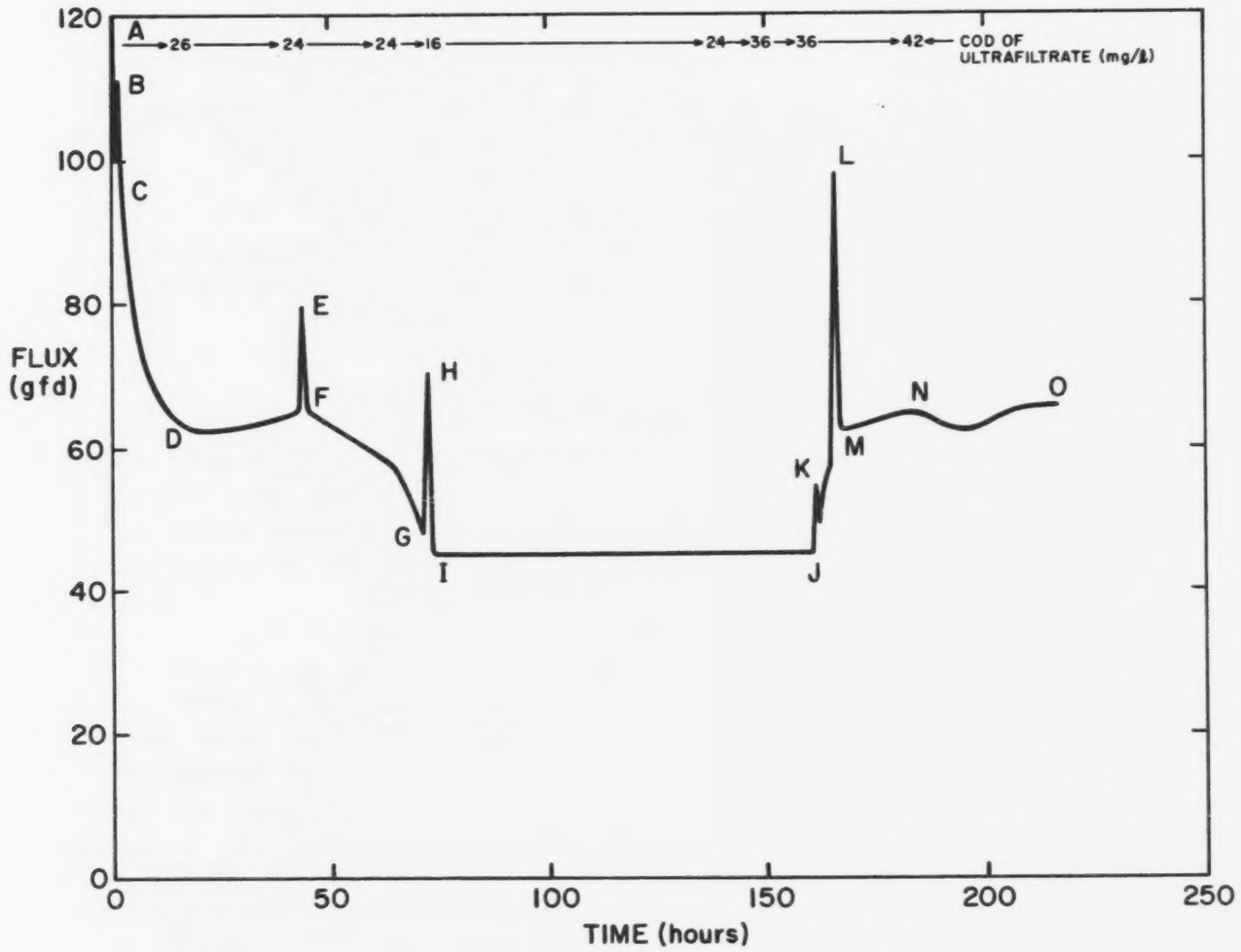
The initial slurry dewatering up to the 10% level is shown. At this level, the flux rate of 87 gfd is comparable to the 75 gfd obtained at the same solids level commencing Run 11-1, although a higher initial fluid velocity of 9.3 ft/sec was employed in this run. Again the system was kept at 26-29°C by keeping the cell immersed in a water bath.

The operating conditions were left unchanged for the next 44 hours during which time the flux appeared to asymptote at the 60-65 gfd level. At this point the fluid velocity was momentarily increased to 11.3 ft/sec to see if a periodic "scouring" would elevate the flux. Although the flux did increase to 80 gfd during the scouring operation. the flux returned to its previous 60-65 gfd value when the original circulate velocity of 9.3 ft/sec was restored. During the next 24 hours the flux further decayed to about 50 gfd at which time the circulation rate was briefly increased, again with no permanent effect on the flux rate.

Operating conditions were left unchanged during the next 3 days during which the flux rate was constant at 45 gfd.

At the 161 hour mark, the cooling bath was removed and the fluid velocity was increased to 11.4 ft/sec and resulted in a substantial flux increase to 98 gfd at a temperature of 43°C.

The fluid velocity was then restored to 9.3 ft/sec while maintaining the temperature at the 43°C level. Under these conditions of elevated temperature, the run was continued through the 216 hour mark (9 days) at which time it was terminated because of a leaking rear seal on the Moyno pump.



TC-1 CELL
MOYNO PUMP
XM-100 MEMBRANE
CHANNEL HEIGHT 0.060 in.
10% SOLIDS

A 5% SOLIDS, V = 6.2 ft/sec
B DEWATERING, V = 7.6 ft/sec
C 10% SOLIDS, RUN BEGINS, ΔP = 12 psi V = 9.3 ft/sec
D ΔP = 13 psi V = 9.3 ft/sec
E ΔP = 16 psi V = 11.3 ft/sec
F ΔP = 13 psi V DECREASED TO 8.2 ft/sec
G V INCREASED TO 10.5 ft/sec
H ΔP = 17.5 psi V = 10.5 ft/sec
I ΔP = 13 psi V = 8.2 ft/sec
J COOLING REMOVED, V = 8.2 ft/sec T = 28°C
K V = 9.3 ft/sec
L ΔP = 20 psi V = 11.3 ft/sec
M ΔP = 14 psi V = 7.6 ft/sec
N ΔP = 16 psi T = 42°C
O RUN ENDED, MOYNO LEAK ΔP = 16 psi

FIGURE 13. LONG TERM DEWATERING RUN 11-2

Note that operations at the elevated temperature and the slightly decreased fluid velocity of 7.6 ft/sec resulted in a flux rate of about 65 gfd.

As in the previous run the COD levels were monitored from time to time and showed no consistent increase or decrease throughout the run.

Effect of Operating Conditions on Flux Rates

Two sets of experiments were carried out to determine the effect of operating variables such as pressure and circulation rate (i.e. shear rate) on flux at various solids levels. In brief, the results showed that at circulation rates in excess of 0.5 gpm, a linear velocity of 5.2 ft/sec in this experiment, the flux rates were directly proportional to the average transmembrane pressure at solids concentrations up to 13%; no runs above 13% were made. At lower circulation rates in the range of 0.04 to 0.42 to 0.06 gpm, 0.42 ft/sec to 0.63 ft/sec in this system, flux rates were found to be relatively independent of pressure, but more dependent on the fluid velocity. In addition, at the lower circulation rates, permeabilities (flux rates per unit pressure) were far less than at the higher circulation rates. These results are consistent with the behavior of other (non-carbon) slurries. Details of the runs are given below.

Circulation Rates in Excess of 0.5 gpm (5.2 ft/sec)

A spent carbon was prepared by treating a secondary effluent with 200 mg/l Aqua Nuchar A and 4 mg/l Primafloc C-7 in the usual manner. The COD of a secondary effluent from which the slurry was derived was 114 mg/l of which 38 mg/l was removable after passage through a sand column.

The resultant 5% slurry was run at three pump speed settings at which the recirculation rate, the pressure drop across the cell (from inlet to outlet on the upstream side), and the transmembrane flux rates were measured. The results are shown in Table X. The slurry was then dewatered to 9% solids and the above procedure repeated. The slurry was finally dewatered to 13% solids

TABLE X

Dewatering as Function of Solids Content for TC-1/Moyno System

XM-100 membrane, channel height: 0.060 in.
Spent carbon slurry prepared at Brockton

Carbon % Solids	Moyno Setting	Circulation Rate gpm	Fluid Velocity ft/sec	Transmembrane Pressure Entering/ Leaving (psi)	Flux gfd	Flux/Average Transmembrane Pressure (gfd/psi)
0	1	0.58	6.2	10/0	133	26.6
(tap water)	2	0.85	9.1	17/0	220	25.9
	3	1.05	11.2	24/0	323	26.9
5	1	0.53	5.7	12/0	85	14.2
	2	0.82	8.8	19/0	123	12.9
	3	0.99	10.6	28/0	165	11.8
9	1	0.58	6.2	16/0	29	3.6
	2	0.87	9.3	25/0	55	4.4
	3	1.05	11.2	35/0	72	4.1
13	1	0.51	5.5	23/0	25	2.2
	2	0.66	7.1	30/0	33	2.2
	3	0.81	8.7	42/0	42	2.0

and the same operating variables were measured. Tap water had been previously run through the system in order to obtain the 0% solids data.

As can be seen from the table the flux per average transmembrane pressure (permeability) at any given solids concentration is fairly constant, indicating that the transmembrane pressure is the variable that controls flux rate at these recirculation rates. These results are also displayed in Figure 14.

Circulation Rates Between 0.04 and 0.05 gpm (0.4-0.6 ft/sec)

A spent carbon slurry was prepared. The COD of the secondary effluent from which the slurry was prepared was 100 mg/l, 33 mg/l of which was removable by sand filtration. The solids content of the settled slurry was 5.6%. To obtain reduced circulation rates at transmembrane pressures comparable to those in the preceding runs, the flow through the TC-1 cell was throttled by valves installed on the inlet and outlet sides of the TC-1 cell. Pressure gauges were installed at the inlet and outlet ports of the TC-1 cell, and the valves adjusted to give a low pressure drop between the cell inlet and outlet to achieve a low circulation rate.

Results of these runs are shown in Table XI. Note that circulation rates were about 1/10 those in Table X, while transmembrane pressure drops were comparable. Flux rates per unit pressure however, fell off sharply.

Comparing the throttled results at 5.8% carbon solids concentration to those at 5% solids in the preceding run, it is immediately apparent that the flux rates per unit pressure drop are about an order of magnitude lower for the throttled runs. These results suggest that in the throttled runs a carbon solids cake of low hydraulic permeability formed on the membrane surface.

Ultrafiltration carried out under such conditions, where the accumulation of ultrafiltered solids on the membrane surface controls the flux rate, generally results in a flux that is relatively insensitive to the transmembrane pressure. Such is the case observed here.

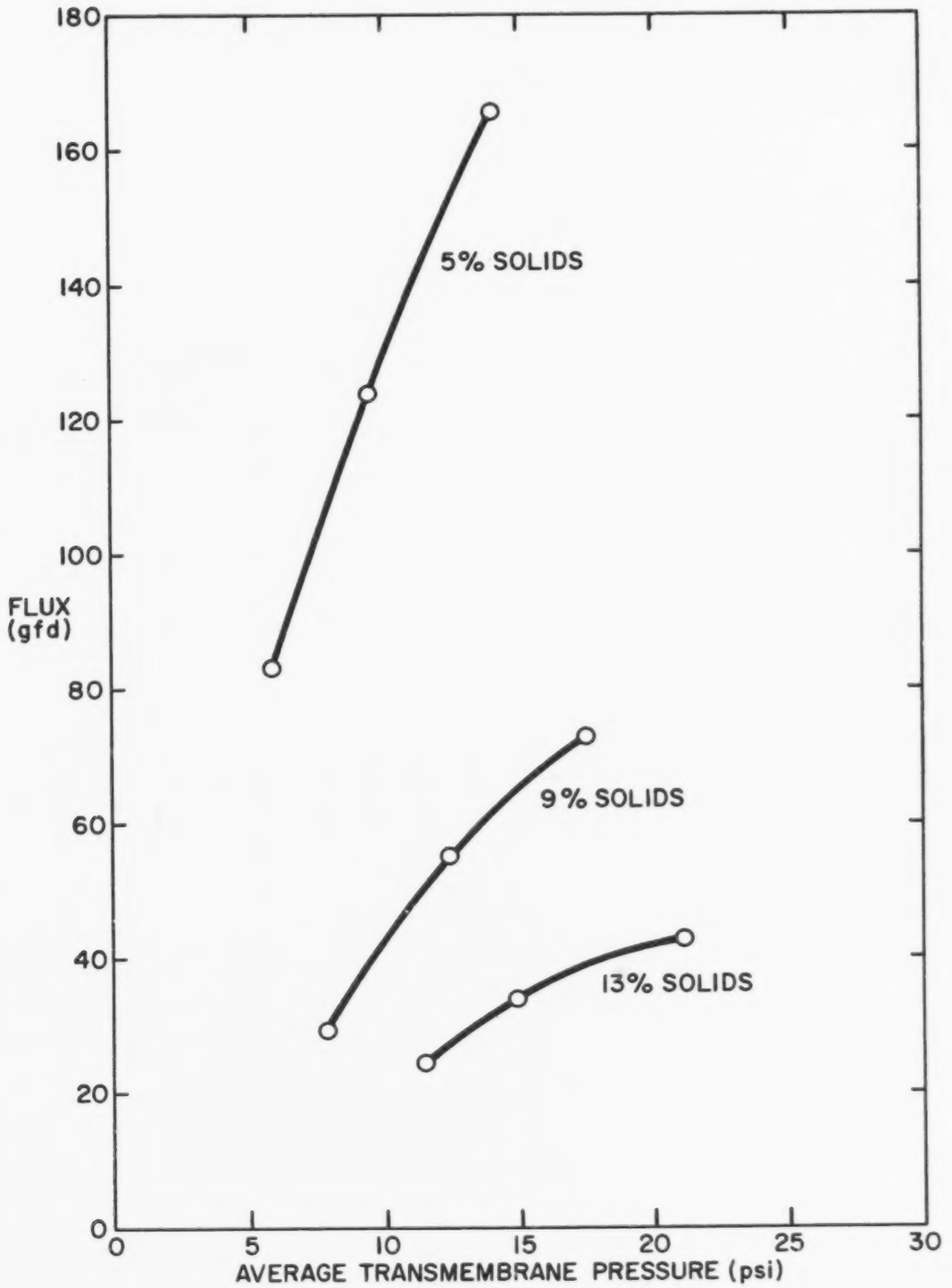


FIGURE 14. EFFECT OF TRANSMEMBRANE PRESSURE ON FLUX

TABLE XI

Dewatering Using Throttled TC-1/Moyno System

XM-100 membrane, channel thickness: 0.06 in.
Spent carbon slurry prepared at Brockton,
5.6% Solids

Moyno Speed Setting	Throttled Circulation Rate (gpm)	Fluid Velocity ft/sec	Transmembrane Pressure Entering/ Leaving (psi)	Flux	Flux/Average Transmembrane Pressure (gal/psi)	Flux Circulation Rate (gfd/gpm)
0.2	0.038	0.41	14/12	19 gfd	1.46	500
1.0	0.053	0.57	17/14	42 gfd	2.70	795
2.0	not measured		26/24	42 gfd	1.68	---
3.0	0.058	0.62	40/38	35 gfd	0.90	605
	(5% solids data from Table X reproduced below for comparison)					
1.0	0.53	5.7	12/0	85 gfd	14.2	160
2.0	0.82	8.8	19/0	123 gfd	12.9	150
3.0	0.99	10.6	28/0	165 gfd	11.8	167

It can be shown on theoretical grounds[1] that a flux rate independent of pressure is the condition to be expected when a layer of retained solids of low hydraulic permeability covers the membrane. The condition is frequently observed when ultrafiltering macromolecular solutions and suspensions and is often referred to as the "gel controlled" region of operation. If the shear rate across the membrane surface is substantially increased, as is the case when the circulation rate is increased (since the shear rate is directly proportional to the circulation rate) then "gel" control can be eliminated and the flux rate becomes directly proportional to the transmembrane pressure as is the case here.

In operating any full scale unit, it is more economical to operate outside of the gel controlled region, i.e. at high circulation rates, since the pumping cost necessary for high circulation rates is less than the additional membrane area required at the low flux rates characteristic of the gel controlled region.

Under the unlikely conditions where pumping costs to circulate the carbon slurries are high, the throttled system may be of some advantage. The flux rates per unit circulation rates for the two modes of operation may be compared by examining the final column in Table XI. The flux per unit circulation rate, otherwise termed dewatering per pass, is about 3 to 5 fold higher for the throttled runs.

RHEOLOGICAL STUDIES

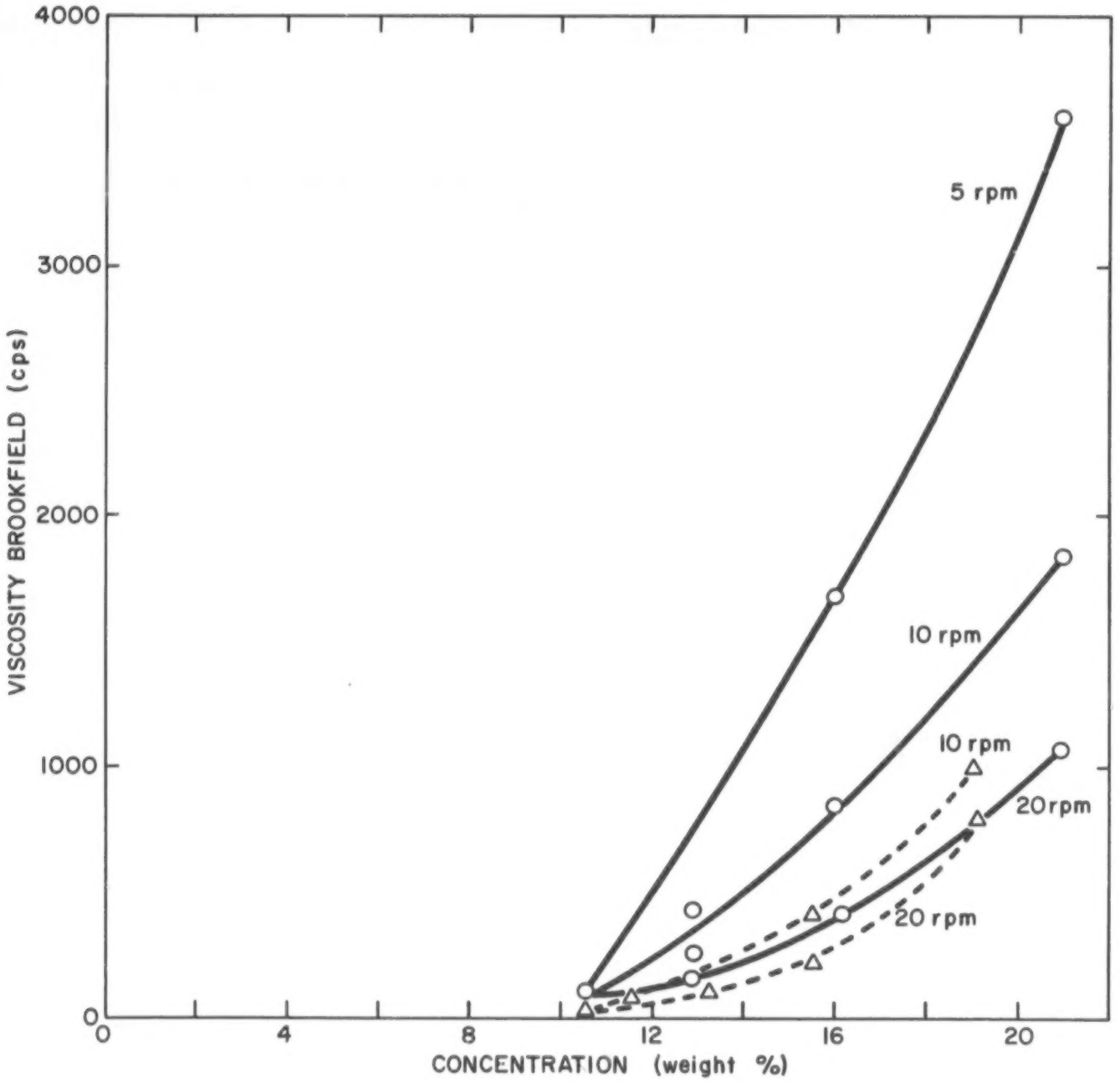
Rheological properties of spent carbon slurries are of interest for two reasons: (1) proper design of plant scale dewatering equipment, especially with regard to pump sizes, channel sizes, etc. and (2) determination of the likelihood that dispersing agents would decrease the viscosity of the slurries thereby enabling them to be dewatered more rapidly or to higher ultimate solids levels.

Most of the rheological studies were carried out using a Brookfield Viscometer. Because the shear rates in the TC-1 cell and in full scale plant equipment are much higher than those encountered with a Brookfield viscometer, some of the pressure/circulation rate data generated in the thin channel cell course of other experiments is analyzed here in rheological terms.

Low Shear Rate Experiments

The effect of carbon solids concentration on Brookfield (Model RVT, Spindle No. 3) viscosity for virgin carbon slurries and a typical Brockton slurry are shown in Figure 15. Note that both virgin and spent carbon viscosities are very low until the 12% carbon concentration is reached, at which point the viscosities climb rapidly. These data would seem to indicate that dewatering much above the 20% level would be difficult because of the viscosity limitation. It should also be noted however, that the viscosity decreases with increasing shear rate; for example the viscosity of the 21% spent carbon sludge decreased from 14,500 centipoise at 5 rpm spindle speed to 4,400 centipoise at 20 rpm spindle speed (fourfold higher shear rate).

Actual shear rate values in a Brookfield decrease from the spindle out into the bulk of the liquid. For the RVT Viscometer, shear rates at the spindle are of the order of 10^{-1} to 10 sec^{-1}.



O - 200 mg/ℓ AQUA NUCHAR A WITH 75 mg/ℓ COD FLOCCULATED WITH PRIMAFLOC C-7
Δ - VIRGIN AQUA NUCHAR A FLOCCULATED WITH PRIMAFLOC C-7
BROOKFIELD RVT SPINDLE NO. 3

FIGURE 15. BROOKFIELD VISCOSITY VS. CONCENTRATION OF CARBON SLURRY

High Shear Rate Experiments

We may use the data in Table X as a source of high shear rate viscosity data.

The TC-1 cell may be used as a viscometer insofar as the channels are of simple rectangular geometry and of uniform dimensions along their entire length.

For a fluid moving in laminar flow through a thin channel the shear stress at the wall of the channel is given by:

$$\tau_w = \frac{h \Delta P}{2L}$$

where h and L are the channel height and length respectively, and ΔP is the pressure drop along the channel. If the liquid is Newtonian, the shear rate is given by:

$$\dot{\gamma} = \frac{6V}{wh^2}$$

where w is the channel width and V is the volumetric flow rate (circulation rate). The viscosity is the ratio of the shear stress to the shear rate.

Inasmuch as carbon slurries are not truly Newtonian, the viscosity values calculated are so-called "apparent viscosities". However the same error is implicit in the Brookfield viscosity values. The apparent viscosity calculations are summarized in Table XII.

It is apparent from the table that the viscosities calculated from this data are far lower than those determined with the Brookfield at the same solids levels. This is a manifestation of the tendency for viscosities of suspensions to decrease markedly with increasing shear rate. It should also be noted that at the high shear rates the viscosities at any given solids concentration are relatively insensitive to changes in shear rate. Since it is known that the viscosities of suspensions decrease with increasing shear rate to an asymptotic value (the asymptotic region often being referred to as the "Upper Newtonian Regime"), the shear rates in the TC-1 unit may

TABLE XII

Apparent Viscosity in TC-1/Moyno System as a Function of Solids Content and Circulation Rate

% Solids	Circulation Rate (ml/sec) Per Channel	Fluid Velocity ft/sec	Pressure Drop psi	Shear Rate $\dot{\gamma} = \frac{6V}{wh^2}$ sec^{-1}	Shear Stress $\tau_w = \frac{h\Delta P}{2L}$ $dynes/cm^2$	Viscosity cp $\tau_w/\dot{\gamma}(100)$
0 (tap water)	18.3	6.2	10	7500	690	9.2
	26.7	9.1	17	10900	1170	10.1
	33.3	11.3	24	13600	1650	12.1
5	16.6	5.6	12	6800	825	12.1
	25.8	8.8	19	10600	1310	12.3
	31.3	10.6	28	12800	1930	15.0
9	18.3	6.2	16	7500	1100	14.7
	26.7	9.1	25	10900	1720	15.8
	33.3	11.3	35	13600	2410	17.6
13	16.0	5.4	23	6600	1585	24.0
	20.8	7.1	30	8500	2070	24.3
	23.7 (est.)	8.0	42	9700 (est.)	2890	28.8

All data were recorded at about 24°C.
TC-1 cell was in cooling bath to hold temperature constant.

V = volumetric flow (cm^3/sec) = circulation rate
w = channel width, 0.635 cm (0.25 in.)
h = channel height, 0.152 cm (0.060 in.)
L = channel length, 76 cm (30.0 in.)
ΔP = pressure drop ($dynes/cm^2$) = 68947 pressure drop (psi)

be sufficiently high to cause complete breakdown of the aggregates into primary particles. If such is the case, then it would be expected that dispersants would be of little value in a TC-1 or other high shear unit. On the other hand, it is conceivable that dispersants could cause aggregate breakdown at even lower shear rates than were used in these experiments.

The calculated viscosity values for water (0% solids) are rather high since water is known to have a viscosity of about 1.0 centipoise at this temperature (independent of shear rate). Unusually high calculated values of viscosities are usually attributable to entrance effects and turbulence. Water moving through the channels at the circulation rates shown, has Reynolds numbers of between 2,000 and 3,500. Such values are in the so-called transition region between laminar and turbulent flow. It is unlikely however that this would account for the tenfold higher viscosity values calculated. These values are more likely due to entrance and exit effects since the fluid is subjected to rapid changes in velocity moving in and out of the cell. In any case, the viscosities of those slurries containing carbon (i.e., 5, 9, and 13%) are sufficiently high such that the Reynolds numbers are in the laminar region.

Also note that at any solids level the apparent viscosity increases with the shear rate. This behavior is consistent with both turbulent flow and entrance effects.

Effect of Dispersants

The effect of dispersant addition on the apparent viscosity of spent carbon slurries was evaluated for two dispersants at low shear rates.

A spent carbon slurry prepared from a 60 mg/l COD secondary effluent was concentrated in the TC-1 unit to 17.5% solids. Various amounts of Tamol 731 dispersant (manufactured by Rohm and Haas, described as a sodium salt of a polymeric carboxylic acid) were added to the slurry and the effect on viscosity determined. The results are shown in Figure 17. Although some reduction in viscosity was observed for the first per cent or so of Tamol, the

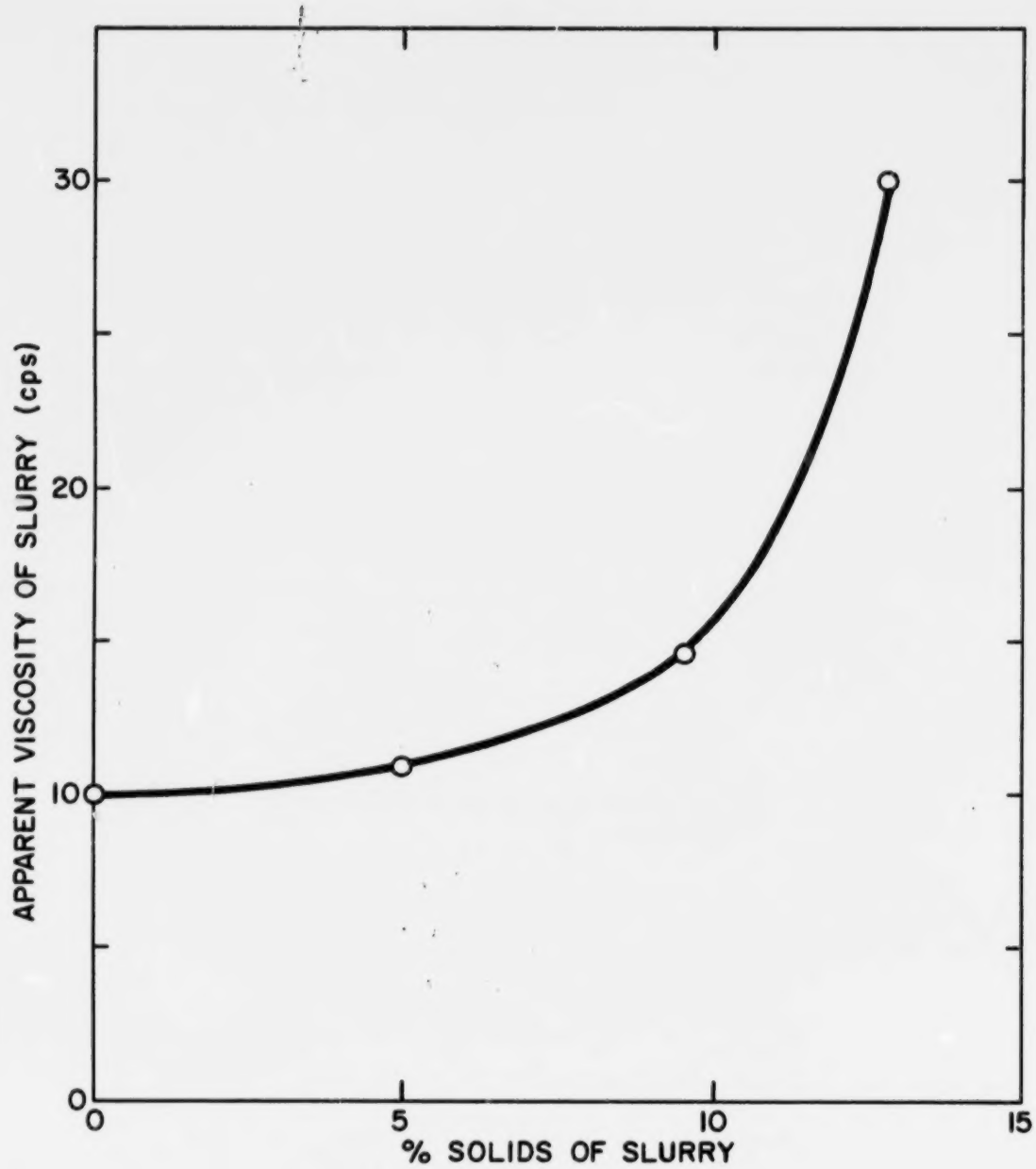
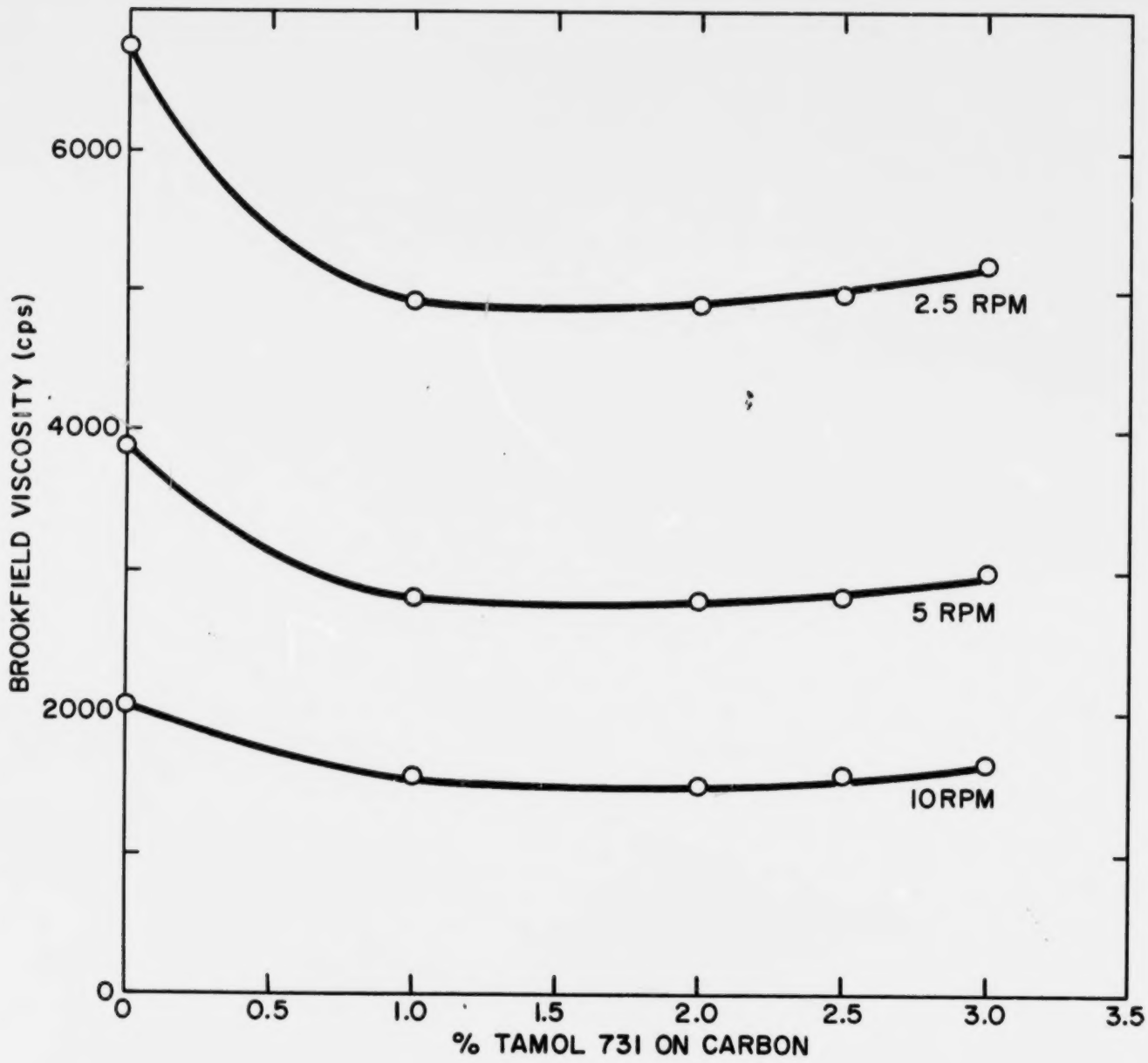


FIGURE 16. APPARENT VISCOSITY OF A CARBON SLURRY AS A FUNCTION OF THE SOLIDS CONTENT



PREPARED FROM 60 mg/ℓ COD BROCKTON SECONDARY EFFLUENT TREATED WITH 200 mg/ℓ AQUA NUCHAR A, FLOCCULATED WITH 5 mg/ℓ PRIMAFLOC C-7, AND SUBSEQUENTLY CONCENTRATED TO 17.5 % SOLIDS

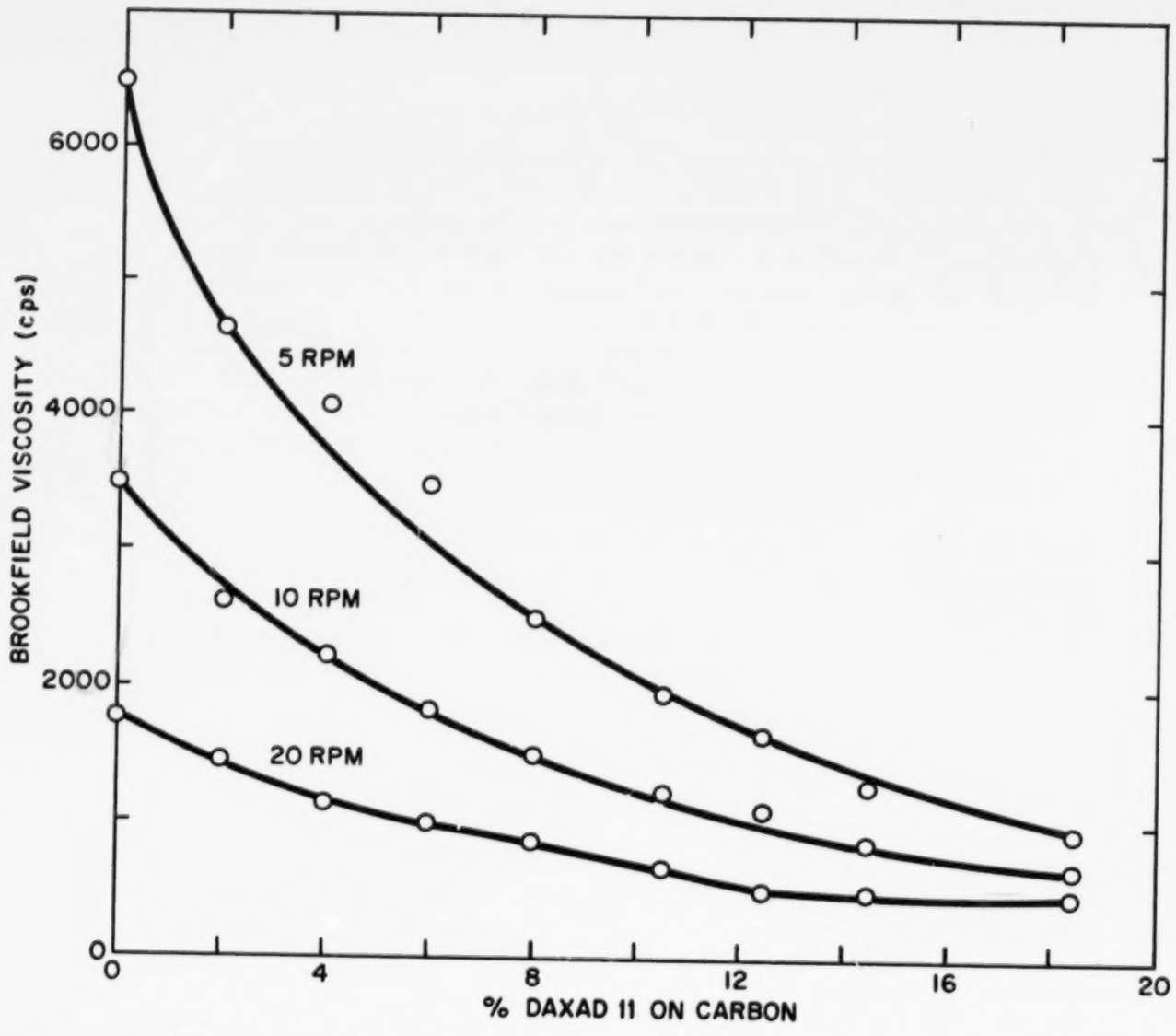
BROOKFIELD RVT SPINDLE NO. 3

FIGURE 17: BROOKFIELD VISCOSITY VS. TAMOL 731 DOSAGE

absolute reduction in viscosity was rather modest and no further reduction was shown at higher Tamol dosages.

In another experiment various amounts of Daxad 11 dispersant (manufactured by W. R. Grace, described as a polymerized sodium salt of a short chain alkyl naphthenate sulfonic acid) were added to Lebanon sample No. 5, which had been concentrated in the TC-1 unit from 10.2% solids level to 17% solids. As shown in Figure 18, the Daxad markedly reduced the viscosity of the slurries particularly at the low shear rates. Although it may be misleading to compare the effectiveness of the dispersants inasmuch as they were evaluated on entirely different sludges (the Brockton material being four times as viscous as the Lebanon material), it would appear that the Daxad is better suited for reducing the viscosity of carbon slurries at low shear rates.

Note in Figure 18 that with increasing spindle speed (shear rate) the effect of the dispersing agent is diminished. This is consistent with the comments in the previous section to the effect that, as the particles are disaggregated by the shear forces themselves, the dispersant becomes proportionately less effective.



(CARBON IS ASSUMED TO HAVE BEEN TREATED WITH 1-2% PRIMAFLOC C-7 AND IS 2/3 OF THE TOTAL SOLIDS)

SPENT CARBON FROM LEBANON CONCENTRATED FROM 10.2 TO 17.0% SOLIDS

BROOKFIELD RVT SPINDLE NO. 3

FIGURE 18. BROOKFIELD VISCOSITY VS. DAXAD 11 DOSAGE

PROPOSED PLANT SCALE PROCESS

General Description

The foregoing results permit the preliminary design of a continuous process to dewater spent carbon slurries by membrane ultrafiltration. In view of the wide change in rheological properties that the carbon slurries undergo with concentration, a two-stage dewatering process is envisioned.

In the first stage, the slurry would be dewatered from its original 5 or 10% level up to about 15 or 17% solids. In this concentration region the viscosity of these slurries is quite low as shown in Figure 16. Conventional pumps and other fluid handling equipment would be adequate to circulate the slurries through the dewatering unit.

In the second stage, the 15 to 17% slurry would be further dewatered to the 20% or above level. In view of the sharply increasing viscosity with concentration of the slurries in this region, the design of the second stage fluid handling equipment is more critical than that of the first. It is likely that the second stage would employ a recirculating loop to permit greater control over the dewatering by smoothing out the effects of variations in the feed slurry solids concentration. The second state would probably have to be provided with control devices to automatically adjust process conditions in response to the changing character of the incoming slurry.

A multiple version of the thin channel spiral cell that was used for most of the dewatering work in this report would not be the proper piece of dewatering equipment for a field or commercial sized unit. A more suitable thin channel device would be that of two concentric tubes, through the narrow annulus of which would flow the carbon slurry. If the outside tube is porous and lined on its inside with membrane, the annulus would be the analogous flow path to the thin channel ultrafiltration cell used in this work. Such tubular devices are currently undergoing prototype evaluation prior to commercial production.

Figure 19 shows the assembly of a tubular cartridge. The sludge would flow longitudinally through the annuli shown in the figure. The membrane surface faces inward and the membrane is supported externally by a braided structure. The solid extruded core has ridges the height of which determines the thin channel space. The ultrafiltrate is collected after having passed through the membrane and braided support. Not shown is the outer cover for the entire cartridge. Figure 20 shows a cross sectional detail of a channel using two different sealing configurations. Not shown in this figure is the braided membrane support.

The XM-100 type would be the recommended membrane since this was the membrane used successfully in this work. The pore sizes in this membrane however are far smaller than necessary to retain carbon particles, the pore sizes being of the order of 100 Å diameter while the particles are several microns in diameter. While it is quite likely that membranes or other filter media with pores as large as 1 micron or possibly larger may be perfectly adequate to completely retain carbon particles, as the pore size of a filter medium approach the size of the carbon particles, the chances of irreversible membrane plugging increase. The filter medium should also be anisotropic in structure, i.e. should have its smallest pores at the carbon contacting surface, in order to reduce the chance of internal plugging of the material.

The experimental results indicate that success in the dewatering of carbon slurries depends less on the characteristics of the membrane than it does on the high shear conditions of thin channel flow as shown in Table X and XI.

Economics

Cost Estimation

The projected costs for dewatering spent carbon via thin channel ultrafiltration are outlined below following the estimation procedures outlined in the Office of Saline Water publication[2].

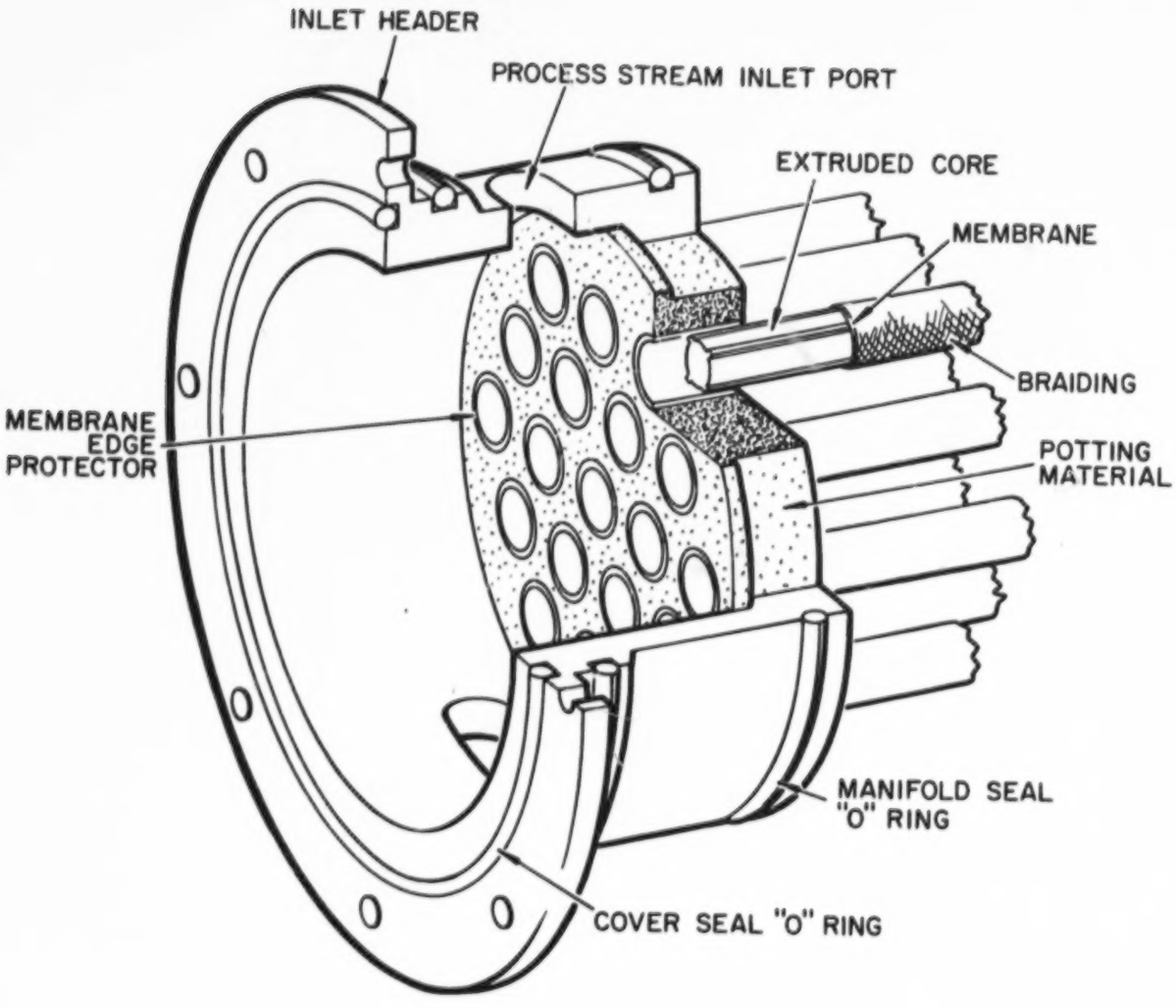


FIGURE 19. DETAIL OF TUBULAR MEMBRANE CARTRIDGE ASSEMBLY

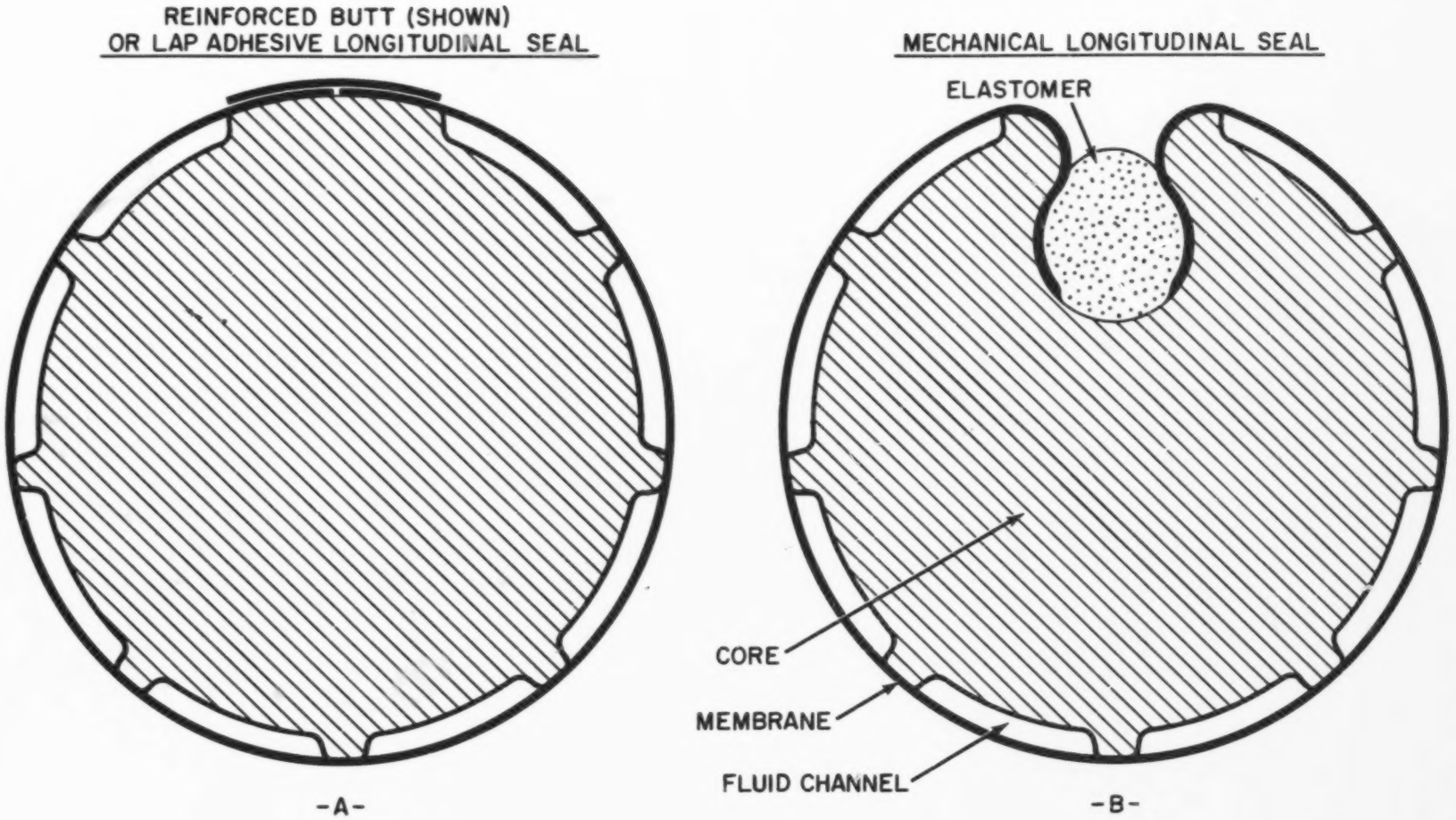


FIGURE 20. ULTRAFILTRATIVE TUBULAR MEMBRANE CONFIGURATION DETAIL

The following figures are given for a ten million gallon per day treatment plant. The dewatering costs are calculated for 5 and 10% spent carbon slurry concentrations, both being dewatered to 20% solids prior to feeding into the reaction furnace. Since this is a small unit to be installed within existing premises, such costs as land acquisition, building construction, raw water supply and product water storage have been omitted. In the Captial Costs a 5 year lifetime has been selected for amortization purposes.

Assuming treatment at the 200 ppm (0.02%) level, a 10 mgd treatment plant would yield 40,000 gallons of 5% slurry or 20,000 gallons of 10% slurry. To concentrate a slurry to 20% carbon, the amount of water to be removed would be 30,000 and 10,000 gallons respectively. The daily weight of carbon to be recovered will be the same in both cases, 16,700 lbs.

The membrane area requirements are estimated assuming two flux rates: 50 and 100 gfd.

Thus:

TABLE 13

Feed Conc. (%)	Ultrafiltrate Volume (gal/day)	Flux Rate (gal/ft^2 day)	Membrane Area Required (ft^2)
5	30,000	50	600
5	30,000	100	300
10	10,000	50	200
10	10,000	100	100

Costs are calculated for each of these four combinations at membrane lifetimes of 1/2, 1, 2, 6, and 12 months.

COSTS

Capital Costs

Spent Carbon Feed (%)	5	5	10	10
Membrane Flux (gfd)	50	100	50	100
A. Essential Plant Costs				
1. Special Equipment				
a. Membrane cell, filters, gauges	$ 4000	$ 2000	$ 1600	$ 1200
b. Membranes ft^2 at $5.00 ft^2	3000	1500	1000	500
2. Standard Engineering Equipment				
a. Air operated diaphragm pump	1200	1200	1200	1200
b. Compressor 2 (1 standby)	2400	2400	1800	1800
c. Moyno pump for second stage or second diaphragm pump	2000 (1200)	2000 (1200)	2000 (1200)	2000 (1200)
B. Other Plant Costs				
1. Installation	$ 1500	$ 1000	$ 800	$ 700
2. Instruments	750	500	400	400
3. Contingencies	1500	1000	1000	1000
4. Engineering	1000	1000	1000	1000
Total Plant Investment	$17350	$12600	$10800	$ 9800
C. Working Capital				
Membrane life				
2 weeks	$16370	$ 8450	$ 5780	$ 3190
1 month	8120	4320	3020	1820
2 months	4588	2550	1840	1226
6 months	2225	1370	1050	830
1 year	1634	995	860	750
Total Capital Costs				
Membrane life				
2 weeks	$33720	$21050	$16600	$13000
1 month	25470	16900	13800	11600
2 months	21940	15150	12650	11000
6 months	19575	13970	11850	10600
1 year	18985	13600	11670	10550

Spent Carbon Feed (%)	5	5	10	10
Membrane Flux (gfd)	50	100	50	100
Capital Cost*/lb of Carbon Processed				
Membrane life				
2 weeks	$0.0012	$0.0008	$0.0006	$0.0005
1 month	0.0009	0.0006	0.0005	0.0004
2 months	0.0008	0.0005	0.0005	0.0004
6 months	0.0007	0.0005	0.0004	0.0004
1 year	0.0007	0.0005	0.0004	0.0004
*5 years x 330 days x 16700 lb/day				
Operating Costs				
A. Essential Operating Costs				
1. Electric Power (typical calculation)				
7.5 HP compressor				
7.5 x .747 x 24 x 2 = 269 KWH				
269 x $0.007 = $1.88/day				
Moyno pump				
38KW/day x .007 = $0.27/day				
Total $2.10 day	$ 2.10	$ 2.10	$ 1.27	$ 1.27
2. Supplies and Maintenance Materials depending on membrane life				
2 weeks 39000/yr	$239.00	$119.50	$ 80.30	$ 40.90
1 month 18000/yr	112.00	56.00	37.90	19.70
2 months 9000/yr	57.60	28.80	19.70	10.60
6 months 3000/yr	21.20	10.60	7.57	4.55
1 year 1500/yr	12.12	4.80	4.55	3.30
3. Operating Labor				
Membrane life				
2 weeks	$ 12.54	$ 6.45	$ 4.41	$ 2.42
1 month	6.19	3.28	2.29	1.36
2 months	3.47	1.92	1.38	0.91
6 months	1.65	1.01	0.77	0.60
1 year	1.19	0.72	0.62	0.54

Spent Carbon Feed (%)	5	5	10	10
Membrane Flux (gfd)	50	100	50	100
4. Maintenance Labor	$ 0.25	$ 0.19	$ 0.16	$ 0.15
5. Payroll Extras				
Membrane life				
2 weeks	$ 1.92	$ 1.00	$ 0.69	$ 0.39
1 month	0.96	0.52	0.37	0.23
2 months	0.56	0.32	0.23	0.16
6 months	0.29	0.18	0.14	0.11
1 year	0.22	0.14	0.12	0.10
Total Essential Operating Costs				
Membrane life				
2 weeks	$255.81	$129.24	$ 86.83	$ 45.14
1 month	121.50	62.09	41.99	22.72
2 months	63.98	33.33	22.74	13.10
6 months	25.49	14.08	9.91	6.69
1 year	15.88	7.95	6.72	5.37
Other Operating Costs				
6. General Overhead and Administrative Overhead				
Membrane life				
2 weeks	$ 4.41	$ 2.29	$ 1.58	$ 0.89
1 month	2.22	1.20	0.85	0.52
2 months	1.28	0.73	0.53	0.37
6 months	0.66	0.41	0.32	0.26
1 year	0.50	0.31	0.27	0.24
7. Amortization*	$ 9.62	$ 7.44	$ 6.56	$ 6.23
8. Taxes and Insurance	$ 1.04	$ 0.76	$ 0.65	$ 0.59

*This equipment is being amortized over a 5 year period at 4% per annum, membranes are excluded from amortization.

Spent Carbon Feed (%)	5	5	10	10
Membrane Flux (gfd)	50	100	50	100
9. Interest on Working Capital				
Membrane life				
2 weeks	$ 1.96	$ 1.01	$ 0.69	$ 0.38
1 month	0.97	0.52	0.36	0.22
2 months	0.55	0.31	0.22	0.15
6 months	0.27	0.16	0.13	0.10
1 year	0.20	0.12	0.10	0.09
Total Operating Costs				
Membrane life				
2 weeks	$272.84	$140.74	$ 96.31	$ 53.23
1 month	135.35	72.01	50.41	30.28
2 months	76.47	42.57	30.70	20.44
6 months	37.08	22.85	17.57	13.87
1 year	27.24	16.58	14.30	12.52
Operating Cost/lb of Carbon Processed				
Membrane life				
2 weeks	$0.0163	$0.0084	$0.0058	$0.0032
1 month	0.0081	0.0043	0.0030	0.0018
2 months	0.0045	0.0025	0.0018	0.0012
6 months	0.0022	0.0014	0.0011	0.0008
1 year	0.0016	0.00099	0.00086	0.00075
Total Cost (Capital Plus Operating) per lb Carbon Processed				
Membrane life				
2 weeks	$0.0175	$0.0092	$0.0064	$0.0037
1 month	0.0090	0.0049	0.0035	0.0022
2 months	0.0053	0.0030	0.0023	0.0016
6 months	0.0029	0.0019	0.0015	0.0012
1 year	0.0023	0.0015	0.0013	0.0011

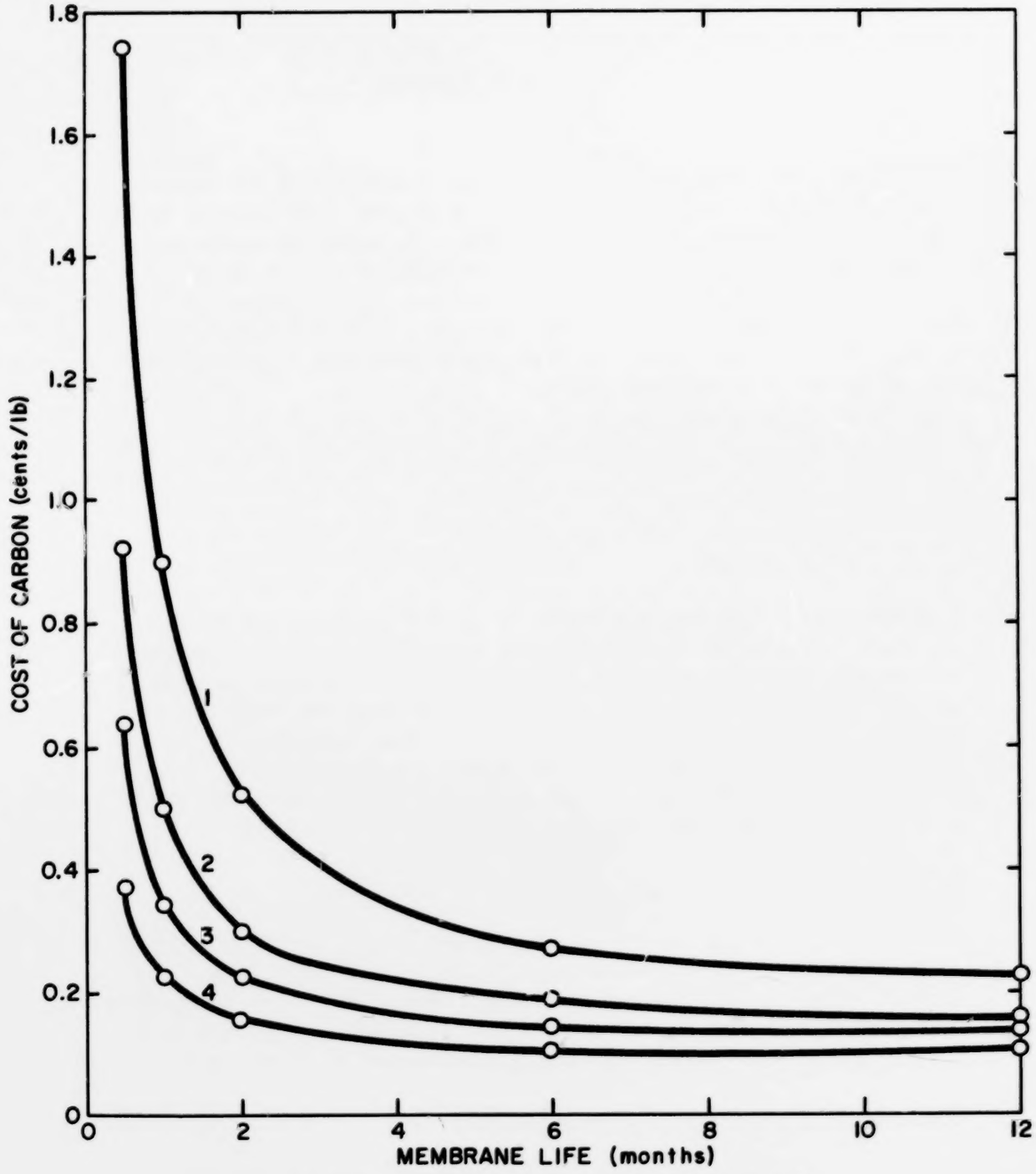
Discussion

This cost estimation is summarized in Figure 21 where the cost of concentrating the spent carbon slurry to the 20+ % level from the 5 and 10% level in cents per pound carbon is expressed as a function of the membrane lifetime (frequency of membrane replacement) at two membrane fluxes: 50 and 100 gfd. As can be seen from the curves, for membrane lifetimes less than 2 or 3 months, the cost of dewatering carbon bares a simple inverse proportionality relationship to membrane flux rate and membrane life.

Reliable estimation of membrane life is made difficult by the unavailability of long term dewatering data. The longest run was of 9 days duration after which the membrane appeared to be in very serviceable condition. Based on these results it can be said with confidence that membrane lifetime is at least 2 weeks, probably 1 to 2 months and possibly 3 months to a half year. Were more robust membranes to be evaluated, the time scale for membrane lifetime might be extended.

The average flux rate most likely to be encountered is probably 100 gfd. Although low solids sludges dewater at higher rates, high solids sludges dewater at lower rates; thus the 100 gfd is a good "average" value. Because membranes show a long term slow loss of flux, the 50 gfd curve may be more appropriate for long membrane lifetimes (in excess of several months).

Thus taking a rather conservative set of conditions, one month membrane life, 50 gfd and 10% solids sludge to be dewatered we arrive at a cost of $0.0035/lb of carbon dewatered.



CURVE	FEED (%)	FLUX (gfd)	AREA ft^2
1	5	50	600
2	5	100	300
3	10	50	200
4	10	100	100

FIGURE 21. ESTIMATED COST TO ULTRAFILTRATIVELY DEWATER SPENT CARBON FROM A 10 MGD TREATMENT PLANT

RECOMMENDATIONS

To further evaluate ultrafiltrative dewatering of spent carbon slurries, a continuous pilot scale dewatering unit should be designed and constructed. A tubular unit such as that described in the section Proposed Plant Scale Process - General Description of about one square foot area would probably be most suitable. For comparison, the laboratory unit used in this program has a membrane area of about 0.1 square feet.

To properly design tubular and larger process units, the following operational information needs yet to be obtained. The items are listed in approximate priority of order:

Membrane Lifetime

A protracted run of at least 1 month's duration should be carried out to determine whether existing polymeric membranes can withstand the sustained scouring action of the powdered carbon. If the lifetime of existing polymeric membranes is inadequate, the feasibility of using more wear resistant inorganic microporous materials should also be evaluated. Candidate materials are porous graphite, silica and sintered metals.

Single Pass Dewatering

Runs should be carried out in which the 5 or 10% carbon slurry feed is dewatered up to the 15% level in a single pass using various channel heights, pressure drops and paths lengths. Similar runs should be carried out between the 15% level up to the 20% and higher level.

Operation at Elevated Temperature

Existing results indicate that operations at elevated temperatures such as 50 and 60°C promote significantly more rapid dewatering. This should be further evaluated in a systematic fashion in order to reduce membrane area requirements.

Effect of Dispersants

Dispersants have been shown to be effective in reducing the viscosity of spent carbon slurries at low shear rates such as those encountered in a Brookfield Viscometer. Their effectiveness in the high shear conditions of the thin channel cell should be determined as a possible method of reducing pumping costs and membrane area requirements.

LIST OF REFERENCES

1. Michaels, A.S., Chemical Engineering Progress, 64 (12) 31 (1968).

2. A Standardized Procedure for Estimating Costs of Saline Water Conversion, March 1966 (OSW Publication).

* U. S. GOVERNMENT PRINTING OFFICE : 1970 O - 408-308